Supporting Information for

Photoredox-catalyzed radical-radical cross coupling of ketyl radicals with unstabilized primary alkyl radicals

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1. General information

1.1 Solvents, reagents, and starting materials

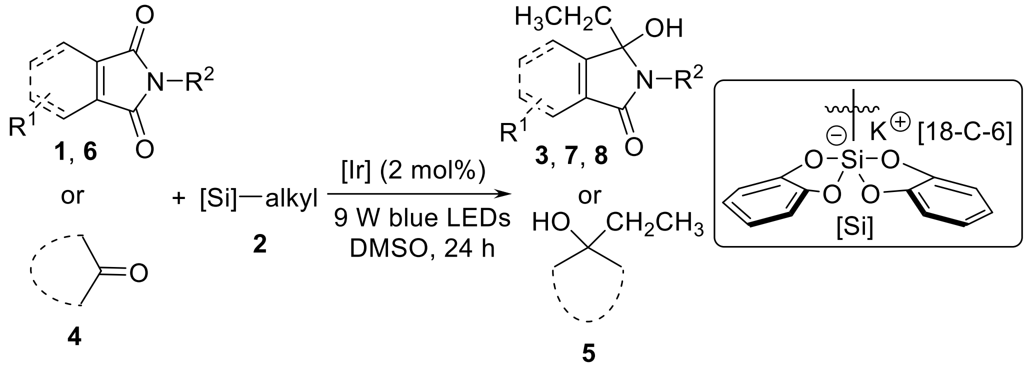
All reactions were carried out under an atmosphere of nitrogen in oven-dried glassware. All photocatalysts, potassium [18-crown-6] bis(catecholato)-alkylsilicates **2**, and β-phthaliminoacrylate methyl ester **6** were reported in our previous literatures.[1] The activated carbonyl compounds **4** and (ω-bromoalkyl)phthalimides **9** were obtained from commercial suppliers and used without further purification. In addition to the commercially available phthalimides **1**, other known *N*-substituted phthalimides can be easily prepared according to reported procedures.[2] Dried solvents were obtained from commercial sources and used without further purification unless otherwise noted.

1.2 Instruments

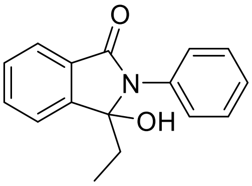
^{1}H NMR, ^{13}C NMR, and ^{19}F NMR nuclear magnetic resonance spectra were recorded on Bruker Avance 500 spectrometer (500 MHz). Chemical shifts were reported in ppm downfield from tetramethylsilane, and calibrated using residue undeuterated solvent ($CDCl_3$ at 7.26 ppm ^{1}H NMR, 77.0 ppm ^{13}C NMR). Spectra were reported as follows: chemical shift (δ ppm), multiplicity (s = singlet, d = doublet, t = triplet, q =quartet, m = multiplet, br = broad), coupling constants (Hz) and integration. High resolution mass spectra (HRMS) were recorded on Agilent 6210 ESI/TOF MS, Thermo Q Exactive Plus, and Waters G2-Xs QTOF mass spectrometers. Analytical thin layer chromatography was performed on Polygram SIL G/UV_{254}plates. Visualization was accomplished with short wave UV light, or $KMnO_4$ staining solutions. Flash column chromatography was performed using silica gel (300-400 mesh) with solvents to use.

1.3 Picture of a typical reaction setup

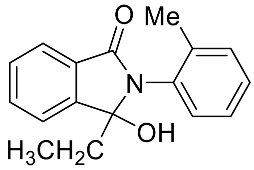
2. General procedure of photocatalysed radical-radical cross coupling reaction of ketyl radical with primary alkyl radical



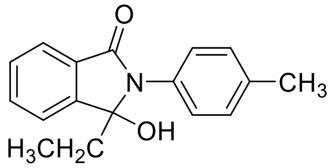
To an oven dried transparent 10 mL Schlenk tube equipped with stirring bar, Ir[dF(CF_3)ppy]$_2$(dtbbpy)PF_6 (4.5 mg, 0.004 mmol, 2 mol %) and *N*-arylphthalimides **1**, **6** or activated ketones **4** (0.2 mmol, 1.0 equiv) were added. Then, potassium [18-crown-6] bis(catecholato)-alkylsilicate **2** (0.4 mmol, 2.0 equiv) was added into the tube. The tube was sealed with a rubber septum. The tube was then charged with degassed DMSO (6.0 mL, 0.033 M) via a syringe under N_2. The tube was irradiated with a 9 W blue LEDs strip spiraled within a bowel for 24 h (cooling with a fan). After the reaction was complete, the reaction solution was diluted with saturated Na_2CO_3 aqueous solution, and was extracted with EtOAc (4 x 5 mL). The organic layer was washed with brine, dried over $MgSO_4$, filtered, and solvent was evaporated to obtain crude product. Flash chromatography over silica gel afforded the product.



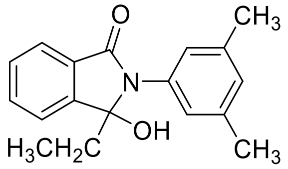
3-Ethyl-3-hydroxy-2-phenylisoindolin-1-one (3a). The product **3a** was obtained in 78% (39.5 mg) yield as a white solid after column chromatography. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.61-7.52 (m, 5H), 7.41-7.32 (m, 3H), 7.28-7.25 (m, 1H), 3.86 (s, 1H), 2.13-2.06 (m, 1H), 2.01-1.94 (m, 1H), 0.45 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 167.0, 146.0, 135.5, 132.8, 131.1, 129.7, 128.8, 126.8, 126.3, 123.7, 121.7, 93.9, 28.8, 7.8. This compound has been reported in the published literature.[3]



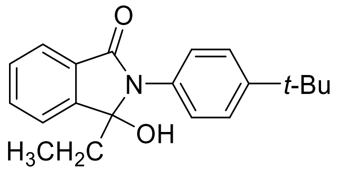
3-Ethyl-3-hydroxy-2-(o-tolyl)isoindolin-1-one (3b). The product **3b** was obtained in 57% (30.5 mg) yield as a white solid after column chromatography. ^{1}H NMR (500 MHz, $CDCl_3$, observed as a mixture of rotamers) δ 7.87 (d, *J* = 7.5 Hz, 0.5H), 7.75 (d, *J* = 7.5 Hz, 0.5H), 7.65-7.60 (m, 1.5H), 7.56-7.45 (m, 2H), 7.36-7.28 (m, 2.5H), 7.20-7.15 (m, 1H), 3.29 (s, 0.5H), 2.78 (s, 0.5H), 2.23 (s, 1.5H), 2.22 (s, 1.5H), 2.21-2.17 (m, 0.5H), 2.16-2.08 (m, 0.5H), 2.02-1.98 (m, 0.5H), 1.86-1.81 (m, 0.5H), 0.88 (t, *J* = 7.5 Hz, 1.5H), 0.81 (t, *J* = 7.4 Hz, 1.5H). ^{13}C NMR (126 MHz, $CDCl_3$, observed as a mixture of rotamers) δ 166.9, 165.8, 146.9, 146.0, 139.1, 137.2, 133.8, 133.1, 132.6, 132.4, 131.5, 131.3, 131.2, 130.9, 129.7(4), 129.6(5), 129.3, 128.8, 128.7, 128.1, 126.7, 126.4, 124.0(4), 123.9(6), 122.5, 121.9, 93.7, 92.8, 30.6, 29.9, 19.1, 18.8, 8.6, 8.4. This compound has been reported in the published literature.[4]



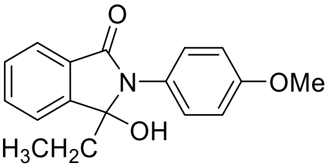
3-Ethyl-3-hydroxy-2-(p-tolyl)isoindolin-1-one (3c). The product **3c** was obtained in 67% (35.8 mg) yield as a white solid after column chromatography. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.58-7.55 (m, 1H), 7.50 (d, *J* = 7.5 Hz, 1H), 7.42 (d, *J* = 7.5 Hz, 1H), 7.37-7.31 (m, 3H), 7.10 (d, *J* = 8.3 Hz, 2H), 4.19 (s, 1H), 2.36 (s, 3H), 2.09-2.02 (m, 1H), 1.96-1.89 (m, 1H), 0.43 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 167.1, 146.1, 136.3, 132.6(4), 132.5(6), 131.0, 129.4, 129.3, 126.1, 123.4, 121.7, 93.8, 28.7, 21.1, 7.7. This compound has been reported in the published literature.[5]



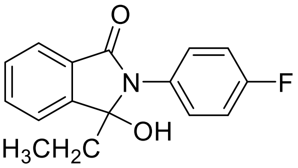
2-(3,5-Dimethylphenyl)-3-ethyl-3-hydroxyisoindolin-1-one (3d). The product **3d** was obtained in 61% (34.3 mg) yield as a white solid after column chromatography. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.63-7.58 (m, 2H), 7.53 (d, *J* = 7.5 Hz, 1H), 7.43-7.40 (m, 1H), 7.15 (s, 2H), 6.92 (s, 1H), 3.53 (s, 1H), 2.30 (s, 6H), 2.11-2.07 (m, 1H), 2.00-1.95 (m, 1H), 0.50 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 167.1, 146.0, 138.3, 135.1, 132.7, 131.2, 129.6, 128.8, 124.3, 123.6, 121.7, 93.7, 28.9, 21.4, 7.9. This compound has been reported in the published literature.[6]



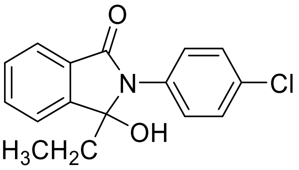
2-(4-(*Tert*-butyl)phenyl)-3-ethyl-3-hydroxyisoindolin-1-one (3e). The product **3e** was obtained in 88% (54.6 mg) yield as a white solid after column chromatography. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.57-7.52 (m, 2H), 7.50-7.49 (m, 1H), 7.45-7.43 (m, 2H), 7.38-7.34 (m, 3H), 4.01 (s, 1H), 2.10-2.03 (m, 1H), 2.00-1.92 (m, 1H), 1.34 (s, 9H), 0.46 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 167.1, 149.5, 146.0, 132.5(8),132.5(7), 131.1, 129.4, 125.9, 125.7, 123.6, 121.7, 93.7, 34.5, 31.3, 28.8, 7.8. HRMS (ESI) $[M+Na]^+$: calculated for $C_{20}H_{23}NO_2Na$: 332.1621, found 332.1626.



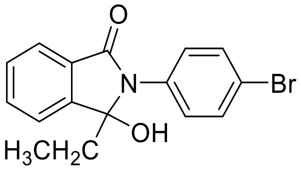
3-Ethyl-3-hydroxy-2-(4-methoxyphenyl)isoindolin-1-one (3f). The product **3f** was obtained in 49% (27.9 mg) yield as a yellow liquid column chromatography. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.56 (t, *J* = 7.4 Hz, 1H), 7.50 (d, *J* = 7.5 Hz, 1H), 7.41-7.36 (m, 3H), 7.32 (t, *J* = 7.4 Hz, 1H), 6.82 (d, *J* = 8.9 Hz, 2H), 4.30 (br, 1H), 3.80 (s, 3H), 2.06-2.00 (m, 1H), 1.92-1.85 (m, 1H), 0.44 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 167.2, 158.1, 146.1, 132.5, 131.0, 129.4, 127.9, 127.8, 123.3, 121.7, 114.0, 93.6, 55.3, 28.8, 7.7. This compound has been reported in the published literature.[6]



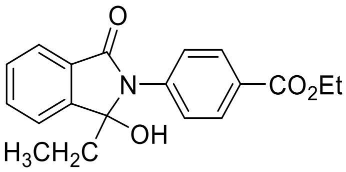
3-Ethyl-2-(4-fluorophenyl)-3-hydroxyisoindolin-1-one (3g). The product **3g** was obtained in 43% (23.2 mg) yield as a white solid column chromatography. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.59 (t, *J* = 7.4 Hz, 1H), 7.53-7.49 (m, 3H), 7.43 (d, *J* = 7.5 Hz, 1H), 7.35 (t, *J* = 7.4 Hz, 1H), 7.00 (t, *J* = 8.6 Hz, 2H), 2.11-2.04 (m, 1H), 1.95-1.88 (m, 1H), 0.42 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 167.1, 161.0 (d, *J* = 239.4 Hz), 146.0, 132.9, 131.3, 130.7, 129.6, 127.8 (d, *J* = 8.8 Hz), 123.5, 121.8, 115.5 (d, *J* = 22.7 Hz), 93.9, 28.7, 7.6. ^{19}F NMR (471 MHz, $CDCl_3$) δ -115.0. HRMS (ESI) $[M+Na]^+$: calculated for $C_{16}H_{14}NO_2FNa$: 294.0906, found 294.0904.



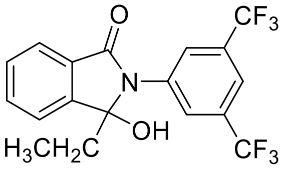
2-(4-Chlorophenyl)-3-ethyl-3-hydroxyisoindolin-1-one (3h). The product **3h** was obtained in 62% (35.6 mg) yield as a white solid after column chromatography. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.62-7.59 (m, 1H), 7.54-7.51 (m, 3H), 7.40 (d, *J* = 7.3 Hz, 1H), 7.34 (t, *J* = 7.4 Hz, 1H), 7.27 (d, *J* = 8.3 Hz, 2H), 4.18 (s, 1H), 2.13-2.06 (m, 1H), 2.00-1.92 (m, 1H), 0.38 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 167.1, 146.0, 134.1, 133.0, 132.0, 130.5, 129.7, 128.8, 126.7, 123.5, 121.7, 94.1, 28.6, 7.6. This compound has been reported in the published literature.[5]



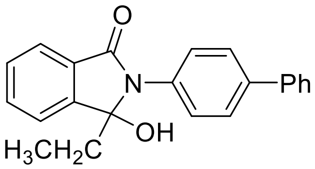
2-(4-Bromophenyl)-3-ethyl-3-hydroxyisoindolin-1-one (3i). The product **3i** was obtained in 69% (45.7 mg) yield as a white solid after column chromatography. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.61-7.58 (m, 1H), 7.52 (d, *J* = 7.5 Hz, 1H), 7.45-7.39 (m, 4H), 7.33-7.30 (m, 2H), 4.39 (s, 1H), 2.11-2.02 (m, 1H), 1.99-1.91 (m, 1H), 0.36 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 167.1, 146.0, 134.6, 133.0, 131.7, 130.4, 129.6, 126.9, 123.4, 121.7, 119.9, 94.2, 28.5, 7.5. This compound has been reported in the published literature.[6]



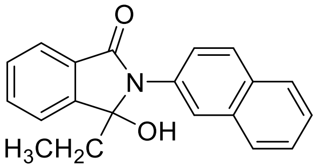
Ethyl 4-(1-ethyl-1-hydroxy-3-oxoisoindolin-2-yl)benzoate (3j). The product **3j** was obtained in 53% (34.2 mg) yield as a yellow solid after column chromatography. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.91 (d, *J* = 8.7 Hz, 2H), 7.70 (d, *J* = 8.7 Hz, 2H), 7.60-7.56 (m, 1H), 7.51 (d, *J* = 7.5 Hz, 1H), 7.40 (d, *J* = 7.5 Hz, 1H), 7.33-7.30 (m, 1H), 4.70 (br, 1H), 4.39-4.32 (m, 2H), 2.12-2.06 (m, 1H), 2.06-2.00 (m, 1H), 1.39 (t, *J* = 7.1 Hz, 3H), 0.33 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 167.2, 166.2, 146.1, 140.0, 133.2, 130.2, 129.9, 129.6, 127.7, 124.2, 123.6, 121.7, 94.5, 61.0, 28.6, 14.3, 7.5. This compound has been reported in the published literature.[5]



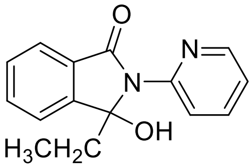
2-(3,5-Bis(trifluoromethyl)phenyl)-3-ethyl-3-hydroxyisoindolin-1-one (3k). The product **3k** was obtained in 37% (29.1 mg) yield as a yellow solid after column chromatography. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.27 (s, 2H), 7.69 (s, 1H), 7.60-7.53 (m, 2H), 7.43 (d, *J* = 7.5 Hz, 1H), 7.28-7.25 (m, 1H), 4.62 (s, 1H), 2.20-2.14 (m, 1H), 2.10-2.01 (m, 1H), 0.32 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 167.3, 145.8, 137.7, 133.8, 131.9 (q, *J* =34.0 Hz), 129.9, 129.4, 123.7(3), 123.6(9), 123.0 (q, *J* =273.4 Hz), 121.8, 119.2(m), 94.9, 28.5, 7.5. ^{19}F NMR (471 MHz, $CDCl_3$) δ -62.9. This compound has been reported in the published literature.[5]



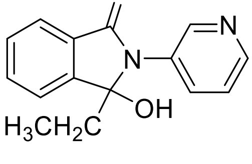
2-([1,1'-Biphenyl]-4-yl)-3-ethyl-3-hydroxyisoindolin-1-one (3l). The product **3l** was obtained in 30% (19.9 mg) yield as a yellow solid after column chromatography. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.63-7.59 (m, 3H), 7.55 (d, *J* = 7.5 Hz, 1H), 7.52-7.49 (m, 4H), 7.44 (d, *J* = 7.5 Hz, 1H), 7.41-7.32 (m, 4H), 4.26 (br, 1H), 2.14-2.09 (m, 1H), 2.07-1.99 (m, 1H), 0.45 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 167.2, 146.1, 140.4, 139.2, 134.7, 132.8, 130.8, 129.6, 128.8, 127.3, 127.2, 127.0, 126.1, 123.5, 121.7, 94.1, 28.8, 7.7. HRMS (ESI) $[M+H]^+$: calculated for $C_{22}H_{20}NO_2$: 330.1494, found 330.1498.



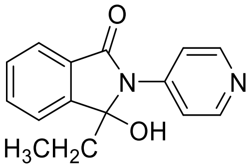
3-Ethyl-3-hydroxy-2-(naphthalen-2-yl)isoindolin-1-one (3m). The product **3m** was obtained in 57% (34.6 mg) yield as a brown solid after column chromatography. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.02 (s, 1H), 7.75 (d, *J* = 8.1 Hz, 1H), 7.68-7.55 (m, 6H), 7.46-7.42 (m, 1H), 7.38 (t, *J* = 7.4 Hz, 2H), 4.06 (br, 1H), 2.15-2.07 (m, 1H), 2.04-1.97 (m, 1H), 0.44 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 167.3, 146.1, 133.3, 133.1, 132.9, 131.9, 130.9, 129.7, 128.3, 128.0, 127.4, 126.0, 125.9, 124.3, 124.0, 123.6, 121.8, 94.3, 28.8, 7.7. This compound has been reported in the published literature.[6]



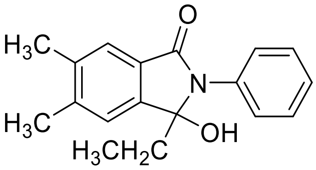
3-Ethyl-3-hydroxy-2-(pyridin-2-yl)isoindolin-1-one (3n). The product **3n** was obtained in 73% (37.1 mg) yield as a yellow liquid after column chromatography. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.41-8.36 (m, 2H), 7.90 (d, *J* = 7.6 Hz, 1H), 7.84-7.81 (m, 1H), 7.69-7.62 (m, 2H), 7.56-7.53 (m, 1H), 7.14-7.12 (m, 1H), 2.64-2.57 (m, 1H), 2.37-2.30 (m, 1H), 0.46 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 166.8, 152.0, 146.5, 145.8, 138.7, 133.4, 131.0, 129.6, 124.0, 121.8, 119.8, 116.6, 95.0, 31.7, 8.4. This compound has been reported in the published literature.[7]



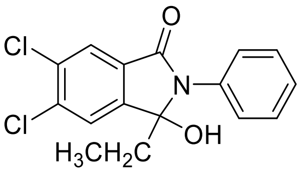
3-Ethyl-3-hydroxy-2-(pyridin-3-yl)isoindolin-1-one (3o). The product **3o** was obtained in 78% (39.7 mg) yield as a yellow solid after column chromatography. ^{1}H NMR (500 MHz, CDCl$_3$) δ 8.83 (d, *J* = 2.0 Hz, 1H), 8.31-8.30 (m, 1H), 8.01-7.99 (m, 1H), 7.69-7.67 (m, 1H), 7.66-7.62 (m, 1H), 7.58-7.56 (m, 1H), 7.47-7.43 (m, 1H), 7.29-7.27 (m, 1H), 5.08 (br, 1H), 2.19-2.12 (m 1H), 2.03-1.94 (m, 1H), 0.46 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR (126 MHz, CDCl$_3$) δ 167.0, 146.8, 146.4, 146.2, 133.3(1), 133.3(0), 133.0, 130.5, 129.8, 123.8, 123.6, 121.9, 94.1, 29.0, 7.6. This compound has been reported in the published literature.[7]



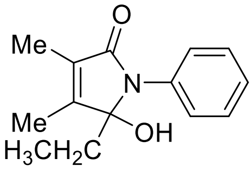
3-Ethyl-3-hydroxy-2-(pyridin-4-yl)isoindolin-1-one (3p). The product **3p** was obtained in 40% (20.4 mg) yield as a white solid after column chromatography. ^{1}H NMR (500 MHz, CDCl$_3$) δ 8.35 (d, *J* = 5.8 Hz, 2H), 7.84 (d, *J* = 6.4 Hz, 2H), 7.68-7.63 (m, 2H), 7.58 (d, *J* = 7.5 Hz, 1H), 7.43 (t, *J* = 7.4 Hz, 1H), 5.24 (br, 1H), 2.31-2.17 (m, 2H), 0.36 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR (126 MHz, CDCl$_3$) δ 167.0, 149.9, 146.3, 144.2, 133.8, 130.1, 129.9, 123.9, 121.7, 117.2, 94.7, 28.6, 7.5. This compound has been reported in the published literature.[7]



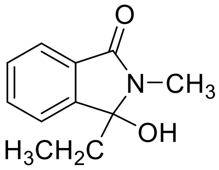
3-Ethyl-3-hydroxy-5,6-dimethyl-2-phenylisoindolin-1-one (3q). The product **3q** was obtained in 23% (13.2 mg) yield as a yellow solid after column chromatography. ^{1}H NMR (500 MHz, CDCl$_3$) δ 7.58 (d, *J* = 7.9 Hz, 2H), 7.44 (s, 1H), 7.35 (t, *J* = 7.8 Hz, 2H), 7.30 (s, 1H), 7.27-7.24 (m, 1H), 3.56 (br, 1H), 2.38 (s, 3H), 2.30 (s, 3H), 2.10-2.04 (m, 1H), 2.01-1.95 (m, 1H), 0.46 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (126 MHz, CDCl$_3$) δ 167.3, 143.9, 142.4, 138.6, 135.8, 129.0, 128.8, 126.6, 126.2, 124.5, 122.6, 93.7, 28.8, 20.6, 20.0, 7.9. HRMS (ESI) [M+Na]$^+$: calculated for $C_{18}H_{19}NO_2Na$: 304.1313, found 304.1315.



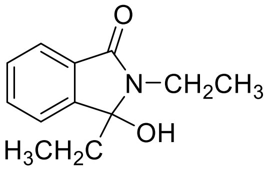
5,6-Dichloro-3-ethyl-3-hydroxy-2-phenylisoindolin-1-one (3r). The product **3r** was obtained in 37% (23.8 mg) yield as a white solid after column chromatography. ^{1}H NMR (500 MHz, CDCl$_3$) (500 MHz, CDCl$_3$) δ 7.64 (s, 1H), δ 7.60 (s, 1H),7.48-7.46 (m, 2H), 7.33-7.30 (m, 2H), 7.29-7.27 (m, 1H), 4.06 (br, 1H), 2.08-2.01 (m, 1H), 1.98-1.91 (m, 1H), 0.47 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (126 MHz, CDCl$_3$) δ 165.1, 145.3, 137.6, 134.7, 134.6, 130.6, 128.9, 127.2, 126.0, 125.3, 124.2, 93.5, 28.7, 7.7. HRMS (ESI) $[M+H]^+$: calculated for $C_{16}H_{14}NO_2Cl_2$: 322.0402, found 322.0408.



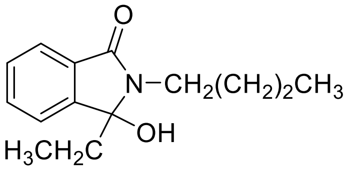
5-Ethyl-5-hydroxy-3,4-dimethyl-1-phenyl-1,5-dihydro-2H-pyrrol-2-one (3s). The product **3s** was obtained in 12% (5.6 mg) yield as a yellow solid after column chromatography. ^1H NMR (500 MHz, $CDCl_3$) δ 7.57-7.55 (m, 2H), 7.33 (t, *J* = 7.9 Hz, 2H), 7.22-7.19 (m, 1H), 2.69 (s, 1H), 1.93 (s, 3H), 1.85 (s, 3H), 1.85-1.74 (m, 2H), 0.50 (t, *J* = 7.5 Hz, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 170.3, 150.1, 136.1, 129.4, 128.8, 125.9, 125.1, 94.5, 26.0, 9.9, 8.5, 7.3. HRMS (ESI) $[M+H]^+$: calculated for $C_{14}H_{18}NO_2$: 232.1338, found 232.1335.



3-Ethyl-3-hydroxy-2-methylisoindolin-1-one (3t). The product **3t** was obtained in 55% (21.1 mg) yield as a yellow solid after column chromatography. ^1H NMR (500 MHz, $CDCl_3$) δ 7.54-7.49 (m, 2H), 7.45-7.44 (m, 1H), 7.36-7.33 (m, 1H), 4.30 (br, 1H), 2.68 (s, 3H), 2.16-2.08 (m, 1H), 2.05-1.97 (m, 1H), 0.39 (t, *J* = 7.5 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 167.5, 146.4, 132.1, 131.2, 129.2, 122.9, 121.7, 91.3, 28.3, 23.0, 7.5. This compound has been reported in the published literature.[6]

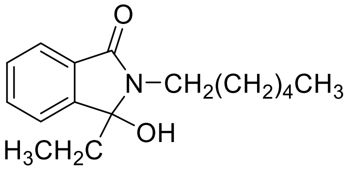


2,3-Diethyl-3-hydroxyisoindolin-1-one (3u). The product **3u** was obtained in 20% (8.2 mg) yield as a colorless liquid after column chromatography. ^1H NMR (500 MHz, $CDCl_3$) δ 7.70 (d, *J* = 7.5 Hz, 1H), 7.57-7.54 (m, 1H), 7.50-7.43 (m, 2H), 3.65-3.57 (m, 1H), 3.32-3.25 (m, 1H), 2.76 (br, 1H), 2.24-2.09 (m, 2H), 1.29 (t, *J* = 7.2 Hz, 3H), 0.52 (t, *J* = 7.5 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 167.3, 146.3, 132.2, 131.8, 129.6, 123.1, 121.5, 92.2, 33.3, 28.9, 14.5, 7.9. This compound has been reported in the published literature.[8]

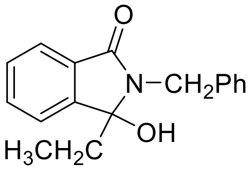


2-Butyl-3-ethyl-3-hydroxyisoindolin-1-one (3v). The product **3v** was obtained in 50% (23.3 mg) yield as a yellow solid after column chromatography. ^1H NMR (500 MHz,

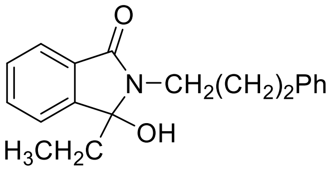
$CDCl_3$) δ 7.66-7.63 (m, 1H), 7.55-7.52 (m, 1H), 7.48 (d, *J* = 7.5 Hz, 1H), 7.44-7.41 (m, 1H), 3.51-3.43 (m, 1H), 3.11 (br, 1H), 3.09-3.02 (m, 1H), 2.24-2.06 (m, 2H), 1.72-1.58 (m, 2H), 1.38-1.33 (m, 2H), 0.93 (t, *J* = 7.4 Hz, 3H), 0.49 (t, *J* = 7.5 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 167.5, 146.3, 132.1, 131.6, 129.4, 123.1, 121.6, 92.1, 38.5, 31.2, 28.9, 20.6, 13.8, 7.8. This compound has been reported in the published literature.[9]



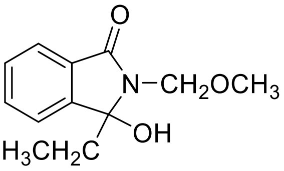
3-Ethyl-2-hexyl-3-hydroxyisoindolin-1-one (3w). The product **3w** was obtained in 39% (20.4 mg) yield as a yellow liquid after column chromatography. ^1H NMR (500 MHz, $CDCl_3$) δ 7.63 (d, *J* = 7.5 Hz, 1H), 7.55-7.52 (m, 1H), 7.48 (d, *J* = 7.4 Hz, 1H), 7.43-7.40 (m, 1H), 3.47-3.41 (m, 1H), 3.25 (s, 1H), 3.06-3.00 (m, 1H), 2.20-2.06 (m, 2H), 1.71-1.58 (m, 2H), 1.35-1.28 (m, 6H), 0.90-0.87 (m, 3H), 0.49 (t, *J* = 7.5 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 167.5, 146.4, 132.1, 131.6, 129.4, 123.1, 121.5, 92.1, 38.7, 31.5, 29.0, 28.9, 27.1, 22.6, 14.0, 7.8. HRMS (ESI) $[M+Na]^+$: calculated for $C_{16}H_{23}NO_2Na$: 284.1626, found 284.1630.



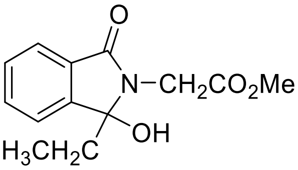
2-Benzyl-3-ethyl-3-hydroxyisoindolin-1-one (3x). The product **3x** was obtained in 50% (26.7 mg) yield as a yellow solid after column chromatography. ^1H NMR (500 MHz, $CDCl_3$) δ 7.70 (d, *J* = 7.5 Hz, 1H), 7.56-7.53 (m, 1H), 7.49-7.42 (m, 4H), 7.28-7.20 (m, 3H), 4.54 (d, *J* = 15.1 Hz, 1H), 4.44 (d, *J* = 15.1 Hz, 1H), 3.30 (br, 1H), 2.13-1.97 (m, 2H), 0.25 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 167.9, 146.5, 138.2, 132.4, 131.2, 129.5, 128.6, 128.4, 127.3, 123.4, 121.7, 92.5, 41.9, 29.2, 7.5. This compound has been reported in the published literature.[10]



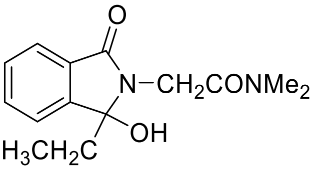
3-Ethyl-3-hydroxy-2-(3-phenylpropyl)isoindolin-1-one (3y). The product **3y** was obtained in 43% (25.4 mg) yield as a yellow liquid after column chromatography. ^1H NMR (500 MHz, $CDCl_3$) δ 7.67 (d, *J* = 7.5 Hz, 1H), 7.55-7.52 (m, 1H), 7.48-7.42 (m, 2H), 7.28-7.25 (m, 2H), 7.20-7.15 (m, 3H), 3.61-3.55 (m, 1H), 3.18-3.12 (m, 1H), 2.69-2.66 (m, 2H), 2.20-1.95 (m, 4H), 0.48 (t, *J* = 7.5 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 167.6, 146.3, 141.6, 132.2, 131.6, 129.6, 128.4, 128.3, 125.9, 123.2, 121.6, 92.1, 38.5, 33.7, 30.5, 28.9, 7.9. HRMS (ESI) $[M+Na]^+$: calculated for $C_{19}H_{21}NO_2Na$: 318.1470, found 318.1469.



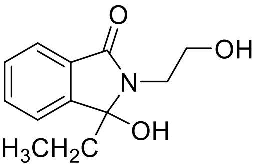
3-Ethyl-3-hydroxy-2-(methoxymethyl)isoindolin-1-one (3z). The product **3z** was obtained in 41% (18.1 mg) yield as a yellow liquid after column chromatography. ^{1}H NMR (500 MHz, CDCl3) δ 7.69 (d, *J* = 7.5 Hz, 1H), 7.63-7.60 (m, 1H), 7.53 (d, *J* = 7.6 Hz, 1H), 7.49-7.46 (m, 1H), 4.89 (d, *J* = 11.1 Hz, 1H), 4.63 (d, *J* = 11.1 Hz, 1H), 3.62 (br, 1H), 3.34 (s, 3H), 2.25-2.15 (m, 2H), 0.55 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR (126 MHz, CDCl3) δ 168.5, 146.6, 133.1, 130.4, 129.6, 123.7, 121.9, 91.8,70.0, 57.0, 30.0, 7.9. This compound has been reported in the published literature.[11]



Methyl 2-(1-ethyl-1-hydroxy-3-oxoisoindolin-2-yl)acetate (3aa). The product **3aa** was obtained in 42% (21.0 mg) yield as a white solid after column chromatography. ^{1}H NMR (500 MHz, CDCl3) δ 7.81 (d, *J* = 7.5 Hz, 1H), 7.63-7.60 (m, 1H), 7.55-7.49 (m, 2H), 4.59 (d, *J* = 17.6 Hz, 1H), 3.86 (d, *J* = 17.6 Hz, 1H), 3.77 (s, 3H), 3.24 (s, 1H), 2.24-2.16 (m, 1H), 2.06-1.99 (m, 1H), 0.55 (t, *J* = 7.5 Hz, 3H). ^{13}C NMR (126 MHz, CDCl3) δ 171.1, 167.7, 146.9, 132.8, 130.7, 129.7, 123.6, 121.9, 91.5, 52.7, 39.7, 28.6, 7.7. This compound has been reported in the published literature.[8]

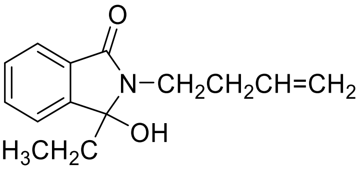


2-(1-Ethyl-1-hydroxy-3-oxoisoindolin-2-yl)-*N*,*N*-dimethylacetamide (3ab). The product **3ab** was obtained in 45% (23.6 mg) yield as a yellow oil after column chromatography. ^{1}H NMR (500 MHz, CDCl3) δ 7.77 (d, *J* = 7.5 Hz, 1H), 7.59-7.56 (m, 1H), 7.53-7.52 (m, 1H), 7.47-7.44 (m, 1H), 5.33 (br, 1H), 4.85 (d, *J* = 16.5 Hz, 1H), 3.77 (d, *J* = 16.6 Hz, 1H), 3.15 (s, 3H), 2.97 (s, 3H), 2.25-2.18 (m, 1H), 2.05-1.98 (m, 1H), 0.53 (t, *J* = 7.5 Hz, 3H). ^{13}C NMR (126 MHz, CDCl3) δ 169.5, 168.4, 147.8, 132.6, 130.5, 129.1, 123.4, 121.7, 91.2, 40.2, 36.8, 36.2, 29.0, 7.7. This compound has been reported in the published literature.[12]

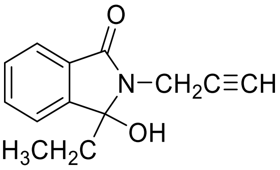


3-Ethyl-3-hydroxy-2-(2-hydroxyethyl)isoindolin-1-one (3ac). The product **3ac** was obtained in 50% (22.3 mg) yield as a yellow oil after column chromatography. ^{1}H

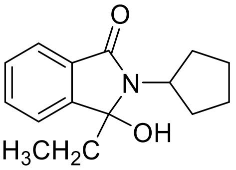
NMR (500 MHz, $CDCl_3$) δ 7.69 (d, *J* = 7.5 Hz, 1H), 7.59-7.56 (m, 1H), 7.50-7.44 (m, 2H), 4.57 (br, 1H), 3.97-3.92 (m, 1H), 3.83-3.76 (m, 2H), 3.28-3.23 (m, 1H), 2.21-2.09 (m, 2H), 0.47 (t, *J* = 7.5 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 168.6, 146.3, 132.6, 131.0, 129.6, 123.3, 121.6, 91.5, 61.8, 41.4, 29.0, 7.7.HRMS (ESI) $[M+Na]^+$: calculated for $C_{12}H_{15}NO_3Na$: 244.0950, found 244.0951.



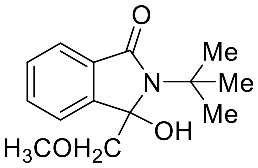
2-(But-3-en-1-yl)-3-ethyl-3-hydroxyisoindolin-1-one (3ad). The product **3ad** was obtained in 31% (14.4 mg) yield as a yellow liquid after column chromatography. ^1H NMR (500 MHz, $CDCl_3$) δ 7.73 (d, *J* = 7.5 Hz, 1H), 7.58-7.55 (m, 1H), 7.50-7.45 (m, 2H), 5.90-5.81 (m, 1H), 5.11-5.03 (m, 2H), 3.76-3.70 (m, 1H), 3.24-3.18 (m, 1H), 2.62 (s, 1H), 2.59-2.43 (m, 2H), 2.24-2.08 (m, 2H), 0.50 (t, *J* = 7.5 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 167.4, 146.3, 136.0, 132.3, 131.5, 129.6, 123.2, 121.6, 116.8, 92.0, 38.1, 33.2, 28.8, 7.8. This compound has been reported in the published literature.[13]



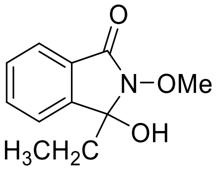
3-Ethyl-3-hydroxy-2-(prop-2-yn-1-yl)isoindolin-1-one (3ae). The product **3ae** was obtained in 56% (24.1 mg) yield as a yellow liquid after column chromatography. ^1H NMR (500 MHz, $CDCl_3$) δ 7.73-7.71 (m, 1H), 7.62-7.59 (m, 1H), 7.54-7.52 (m, 1H), 7.49-7.46 (m, 1H), 4.18-4.02 (m, 2H), 3.13 (br, 1H), 2.28-2.19 (m, 2H), 2.16 (t, *J* = 2.5 Hz, 1H), 0.57 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 166.8, 146.4, 132.7, 130.9, 129.7, 123.5, 121.8, 92.0, 78.9, 70.7, 29.0, 26.8, 8.1. This compound has been reported in the published literature.[8]



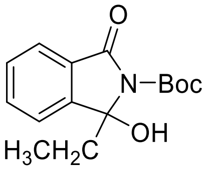
2-Cyclopentyl-3-ethyl-3-hydroxyisoindolin-1-one (3af). The product **3af** was obtained in 60% (29.4 mg) yield as a white solid after column chromatography. ^1H NMR (500 MHz, $CDCl_3$) δ 7.72-7.70 (m, 1H), 7.55-7.52 (m, 1H), 7.47-7.44 (m, 2H), 3.88-3.81 (m, 1H), 2.34-2.25 (m, 2H), 2.21-2.14 (m, 1H), 2.13-2.07 (m, 1H), 2.01-1.90 (m, 2H), 1.88-1.79 (m, 2H),1.64-1.53 (m, 2H), 0.55 (t, *J* = 7.5 Hz, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 166.5, 145.7, 132.6, 131.9, 129.6, 122.8, 121.5, 92.3, 52.2, 30.0, 29.1, 29.0, 25.1, 25.0, 8.0. HRMS (ESI) $[M+H]^+$: calculated for $C_{15}H_{20}NO_2$: 246.1494, found 246.1496.



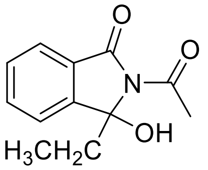
2-(*tert*-Butyl)-3-hydroxy-3-(methoxymethyl)isoindolin-1-one (3ag). The product **3ag** was obtained in 38% (19.1 mg) yield as a white solid after column chromatography. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.65 (d, *J* = 7.4 Hz, 1H), 7.53-7.46 (m, 2H), 7.44-7.41 (m, 1H), 4.01 (d, *J* = 10.0 Hz, 1H), 3.75 (d, *J* = 9.9 Hz, 1H), 3.27 (s, 3H), 1.64 (s, 9H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 168.4, 146.9, 131.9, 131.8, 129.5, 122.6, 121.5, 92.0, 76.0, 59.3, 56.4, 29.1. HRMS (ESI) [M+Na]$^+$: calculated for $C_{14}H_{19}NO_3Na$: 272.1263, found 272.1269.



3-Ethyl-3-hydroxy-2-methoxyisoindolin-1-one (3ah). The product **3ah** was obtained in 42% (17.4 mg) yield as a brown liquid after column chromatography. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.73-7.71 (m, 1H), 7.61-7.58 (m, 1H), 7.48-7.45 (m, 2H), 4.04 (s, 3H), 3.47 (br, 1H), 2.31-2.24 (m, 1H), 2.17-2.10 (m, 1H), 0.55 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 164.4, 143.6, 133.1, 129.7, 129.0, 123.4, 121.9, 92.1, 65.7, 28.9, 7.9. This compound has been reported in the published literature.[7]

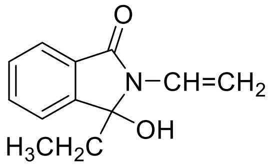


***tert*-Btyl 1-ethyl-1-hydroxy-3-oxoisoindoline-2-carboxylate (3ai).** The product **3ai** was obtained in 36% (20.0 mg) yield as a yellow liquid after column chromatography. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.85 (d, *J* = 7.6 Hz, 1H), 7.70-7.67 (m, 1H), 7.57-7.53 (m, 2H), 4.55 (s, 1H), 2.61-2.53 (m, 1H), 2.30-2.23 (m, 1H), 1.62 (s, 9H), 0.58 (t, *J* = 7.5 Hz, 3H).^{13}C NMR (126 MHz, $CDCl_3$) δ 165.1, 151.3, 145.2, 134.2, 130.1, 124.5, 122.2, 92.9, 84.1, 32.2, 28.2, 8.5. HRMS (ESI) [M+Na]$^+$: calculated for $C_{15}H_{19}NO_4Na$: 300.1212, found 300.1208.

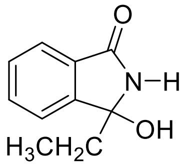


2-Acetyl-3-ethyl-3-hydroxyisoindolin-1-one (3aj). The product **3aj** was obtained in 58% (25.6 mg) yield as a yellow oil after column chromatography. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.88 (d, *J* = 7.6 Hz, 1H), 7.74-7.71 (m, 1H), 7.61-7.56 (m, 2H), 4.86 (br, 1H), 2.68 (s, 3H), 2.66-2.60 (m, 1H), 2.30-2.23 (m, 1H), 0.53 (t, *J* = 7.5 Hz, 3H).

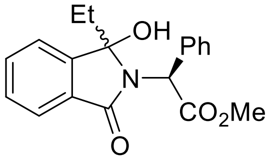
^{13}C NMR (126 MHz, CDCl$_3$) δ 173.2, 166.9, 145.8, 134.9, 130.2, 129.6, 124.7, 122.3, 93.8, 32.1, 25.9, 8.6. HRMS (ESI) [M+Na]$^+$: calculated for $C_{12}H_{13}NO_3Na$: 242.0793, found 242.0797.



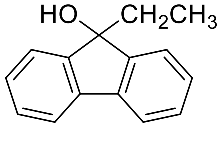
3-Ethyl-3-hydroxy-2-vinylisoindolin-1-one (3ak). The product **3ak** was obtained in 21% (8.6 mg) yield as a yellow liquid after column chromatography. ^{1}H NMR (500 MHz, CDCl$_3$) δ 7.69 (d, *J* = 7.5 Hz, 1H), 7.63-7.60 (m, 1H), 7.55 (d, *J* = 7.6 Hz, 1H), 7.49-7.46 (m, 1H), 6.79 (dd, J_1= 16.5 Hz, J_2= 10.0 Hz, 1H), 5.40 (d, *J* = 16.5 Hz, 1H), 4.70 (d, *J* = 9.9 Hz, 1H), 3.27 (br, 1H), 2.49-2.42 (m, 1H), 2.23-2.16 (m, 1H), 0.42 (t, *J* = 7.5 Hz, 3H). ^{13}C NMR (126 MHz, CDCl$_3$) δ 165.6, 146.5, 133.2, 130.3, 129.9, 125.9, 123.7, 121.7, 99.4, 92.7, 28.6, 7.5. This compound has been reported in the published literature.[14]



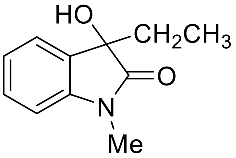
3-Ethyl-3-hydroxyisoindolin-1-one (3al). The product **3al** was obtained in 16% (5.7 mg) yield as a white solid after column chromatography. ^{1}H NMR (500 MHz, CDCl$_3$) δ 7.65 (d, *J* = 7.5 Hz, 1H), 7.61-7.58 (m, 1H), 7.54-7.51 (m, 1H), 7.47-7.44 (m, 1H), 6.61 (br, 1H), 3.29 (s, 1H), 2.16-2.04 (m, 2H), 0.85 (t, *J* = 7.5 Hz, 3H). ^{13}C NMR (126 MHz, CDCl$_3$) δ 169.1, 148.2, 132.9, 130.7, 129.7, 123.6, 121.9, 88.7, 31.5, 8.1. This compound has been reported in the published literature.[6]



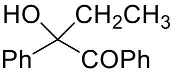
(1-Ethyl-1-hydroxy-3-oxo1-1,3-dihydroisoindol-2-yl) phenyl acetic acid methyl ester (**3am**). The major diastereoisomer of **3am** was obtained in 46% (30.0 mg) yield as a colorless solid after column chromatography.^{1}H NMR (500 MHz, CDCl$_3$) δ 7.81 (d, *J* = 8.2 Hz, 1H), 7.62-7.58 (m, 2H), 7.52-7.50 (m, 2H), 7.39-7.32 (m, 4H), 5.19 (s, 1H), 3.74 (s, 3H), 3.43 (s, 1H), 2.14-2.09 (m, 1H), 1.93-1.89 (m, 1H), 0.20 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR (126 MHz, CDCl$_3$) δ 170.6, 167.7, 146.4, 136.1, 132.7, 131.0, 129.7, 129.6, 128.3, 128.1, 123.7, 121.9, 92.3, 57.4, 53.2, 29.2, 7.5. This compound has been reported in the published literature.[15]



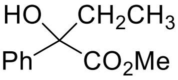
9-Ethyl-9H-fluoren-9-ol (5a). The product **5a** was obtained in 35% (14.7 mg) yield as a white solid after column chromatography. ^{1}H NMR (500 MHz, CDCl$_3$) δ 7.62 (d, *J* = 7.4 Hz, 2H), 7.51 (d, *J* = 7.4 Hz, 2H), 7.38-7.35 (m, 2H), 7.32-7.29 (m, 2H), 2.22-2.17 (m, 2H), 2.06 (s, 1H), 0.56 (t, *J* = 7.5 Hz, 3H). ^{13}C NMR (126 MHz, CDCl$_3$) δ 148.4, 139.7, 128.9, 127.9, 123.5, 119.9, 83.1, 32.3, 8.4. This compound has been reported in the published literature.[16]



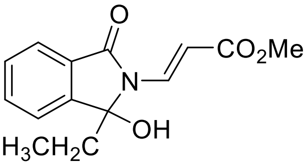
3-Ethyl-3-hydroxy-1-methylindolin-2-one (5b). The product **5b** was obtained in 38% (14.6 mg) yield as a white solid after column chromatography. ^{1}H NMR (500 MHz, CDCl$_3$) δ 7.38 (d, *J* = 7.3 Hz, 1H), 7.35-7.32 (m, 1H), 7.12-7.09 (m, 1H), 6.84 (d, *J* = 7.8 Hz, 1H), 3.20 (s, 3H), 2.04-1.94 (m, 2H), 0.76 (t, *J* = 7.5 Hz, 3H). ^{13}C NMR (126 MHz, CDCl$_3$) δ 178.2, 143.6, 129.7, 129.6, 123.8, 123.1, 108.4, 77.2, 31.6, 26.1, 7.5. This compound has been reported in the published literature.[17]



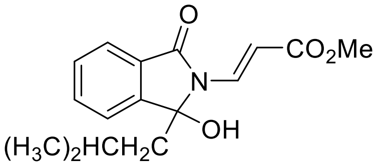
2-Hydroxy-1,2-diphenylbutan-1-one (5c). The product **5c** was obtained in 70% (33.6 mg) yield as a white solid after column chromatography. ^{1}H NMR (500 MHz, CDCl$_3$) δ 7.66-7.64 (m, 2H), 7.47-7.44 (m, 3H), 7.40-7.37 (m, 2H), 7.33-7.28 (m, 3H), 4.72 (br, 1H), 2.41 (q, *J* = 7.5 Hz, 2H), 0.85 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (126 MHz, CDCl$_3$) δ 202.1, 142.2, 134.2, 132.9, 129.8, 128.9, 128.3, 128.1, 126.3, 81.9, 30.5, 7.4. This compound has been reported in the published literature.[18]



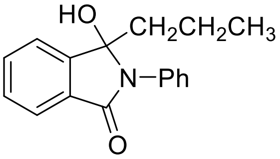
Methyl 2-hydroxy-2-phenylbutanoate (5d). The product **5d** was obtained in 58% (22.6 mg) yield as a colorless liquid after column chromatography. ^{1}H NMR (500 MHz, CDCl$_3$) δ 7.60-7.58 (m, 2H), 7.37-7.34 (m, 2H), 7.30-7.27 (m, 1H), 3.79 (s, 3H), 3.72 (s, 1H), 2.27-2.20 (m, 1H), 2.07-2.00 (m, 1H), 0.92 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (126 MHz, CDCl$_3$) δ 175.8, 141.7, 128.2, 127.7, 125.6, 78.8, 53.2, 32.6, 8.0. This compound has been reported in the published literature.[19]



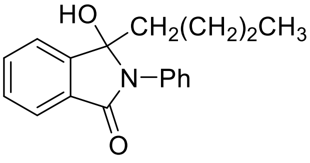
Methyl (*E*)-3-(1-ethyl-1-hydroxy-3-oxoisoindolin-2-yl)acrylate(7a). The product **7a** was obtained in 67% (35.0 mg) yield as a brown solid after column chromatography. ^{1}H NMR (500 MHz, CDCl$_3$) δ 7.71-7.65 (m, 3H), 7.58 (d, *J* = 7.6 Hz, 1H), 7.47 (t, *J* = 7.4 Hz, 1H), 6.17 (d, *J* = 14.7 Hz, 1H), 4.70 (br, 1H), 3.67 (s, 3H), 2.39-2.31 (m, 1H), 2.24-2.16 (m, 1H), 0.40 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR (126 MHz, CDCl$_3$) δ 168.5, 166.3, 146.8, 134.4, 134.2, 130.1, 129.1, 124.2, 122.1, 103.0, 92.9, 51.4, 29.4, 7.5. HRMS (ESI) $[M+Na]^+$: calculated for $C_{14}H_{15}NO_4Na$: 284.0899, found 284.0898.



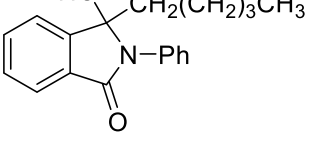
Methyl (*E*)-3-(1-hydroxy-1-isobutyl-3-oxoisoindolin-2-yl)acrylate (7b). The product **7b** was obtained in 77% (44.6 mg) yield as a yellow solid after column chromatography. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.74-7.71 (m, 2H), 7.66 (t, *J* = 7.4 Hz, 1H), 7.60 (d, *J* = 7.5 Hz, 1H), 7.49 (t, *J* = 7.4 Hz, 1H), 6.22 (d, *J* = 14.7 Hz, 1H), 4.14 (br, 1H), 3.70 (s, 3H), 2.33-2.29 (m, 1H), 2.15-2.11 (m, 1H), 1.09-1.03 (m, 1H), 0.70 (d, *J* = 6.7 Hz, 3H), 0.47 (d, *J* = 6.6 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 168.4, 166.3, 147.4, 134.8, 134.0, 130.2, 128.9, 124.4, 122.5, 103.3, 92.0, 51.4, 44.5, 24.0, 23.5, 23.4. HRMS (ESI) $[M+Na]^+$: calculated for $C_{16}H_{19}NO_4Na$: 312.1212, found 312.1210. Single crystal of **7b** was recrystallized from mixed solvents of ethanol and petroleumether by slow evaporation at room temperature.



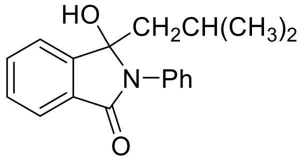
3-Hydroxy-2-phenyl-3-propylisoindolin-1-one (8a). The product **8a** was obtained in 80% (42.8 mg) yield as a white solid after column chromatography. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.61-7.58 (m, 2H), 7.55-7.52 (m, 3H), 7.42-7.39 (m, 1H), 7.37-7.34 (m, 2H), 7.29-7.28 (m, 1H), 3.72 (s, 1H), 2.05-1.99 (m, 1H), 1.95-1.89 (m, 1H), 1.04-0.95 (m, 1H), 0.74-0.67 (m, 1H),0.65 (t, *J* = 7.0 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 167.0, 146.4, 135.5, 132.8, 130.9, 129.6, 128.8, 126.8, 126.4, 123.7, 121.8, 93.2, 38.0, 16.8, 13.7. This compound has been reported in the published literature.[3]



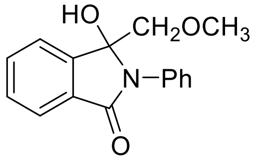
3-Butyl-3-hydroxy-2-phenylisoindolin-1-one (8b). The product **8b** was obtained in 80% (45.0 mg) yield as a yellow liquid after column chromatography. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.66 (d, *J* = 7.5 Hz, 1H), 7.63-7.59 (m, 1H), 7.57-7.54 (m, 3H), 7.45-7.42 (m, 1H), 7.37 (t, *J* = 7.8 Hz, 2H), 7.29 (t, *J* = 7.5 Hz, 1H), 3.47 (br, 1H), 2.09-2.03 (m, 1H), 1.98-1.92 (m, 1H), 1.10-1.03 (m, 2H),0.98-0.85 (m, 2H), 0.65 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 166.9, 146.3, 135.5, 132.8, 130.9, 129.7, 128.8, 126.8, 126.4, 123.8, 121.7, 93.2, 35.5, 25.4, 22.2, 13.6. HRMS (ESI) $[M+H]^+$: calculated for $C_{18}H_{20}NO_2$: 282.1489, found 282.1484.



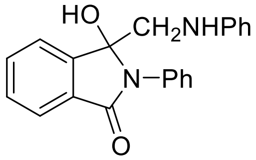
3-Hydroxy-3-pentyl-2-phenylisoindolin-1-one (8c). The product **8c** was obtained in 75% (44.3 mg) yield as a yellow solid after column chromatography. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.59-7.56 (m, 1H), 7.53-7.50 (m, 4H), 7.38-7.31 (m, 3H), 7.27-7.24 (m, 1H), 4.05 (br, 1H), 2.05-1.98 (m, 1H), 1.93-1.87 (m, 1H), 1.05-0.84 (m, 6H), 0.67 (t, *J* = 7.0 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 167.0, 146.5, 135.4, 132.7, 130.8, 129.5, 128.7, 126.7, 126.3, 123.6, 121.7, 93.3, 35.6, 31.2, 22.8, 22.0, 13.7. HRMS (ESI) $[M+H]^+$: calculated for $C_{19}H_{22}NO_2$: 296.1645, found 296.1648.



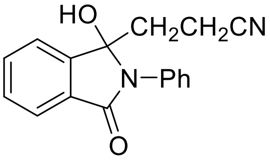
3-Hydroxy-3-isobutyl-2-phenylisoindolin-1-one (8d). The product **8d** was obtained in 74% (41.6 mg) yield as a yellow solid after column chromatography. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.58-7.55 (m, 5H), 7.40-7.36 (m, 1H), 7.34-7.31 (m, 2H), 7.26-7.23 (m, 1H), 3.81 (s, 1H), 2.01-1.92 (m, 2H), 1.28-1.25 (m, 1H), 0.55 (d, *J* = 6.8 Hz, 3H), 0.39 (d, *J* = 6.6 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 166.9, 146.7, 135.7, 132.5, 130.6, 129.6, 128.7, 126.5, 125.9, 123.7, 122.5, 93.3, 44.2, 24.0, 23.7, 23.2. HRMS (ESI) $[M+H]^+$: calculated for $C_{18}H_{20}NO_2$: 282.1489, found 282.1484.



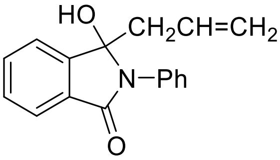
3-Hydroxy-3-(methoxymethyl)-2-phenylisoindolin-1-one (8e). The product **8e** was obtained in 71% (38.2 mg) yield as a yellow solid after column chromatography. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.65-7.63 (m, 2H), 7.61-7.58 (m, 1H), 7.46-7.43 (m, 3H), 7.40-7.37 (m, 2H), 7.35-7.31 (m, 1H), 3.71 (d, *J* = 9.8 Hz, 1H), 3.46 (d, *J* = 9.8 Hz, 1H), 3.14 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 167.4, 145.5, 135.1, 132.6, 131.2, 129.9, 128.9, 127.9, 127.5, 123.6, 122.3, 90.4, 73.7, 59.2. HRMS (ESI) $[M+H]^+$: calculated for $C_{16}H_{16}NO_3$: 270.1125, found 270.1129.



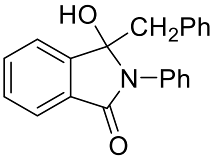
3-Hydroxy-2-phenyl-3-((phenylamino)methyl)isoindolin-1-one (8f). The product **8f** was obtained in 80% (52.9 mg) yield as a brown liquid after column chromatography. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.67 (d, *J* = 7.6 Hz, 1H), 7.58-7.54 (m, 1H), 7.50 (d, *J* = 7.5 Hz, 1H), 7.44-7.43 (m, 2H), 7.40-7.37 (m, 1H), 7.35-7.31 (m, 3H), 7.06-7.03 (m, 2H), 6.66 (t, *J* = 7.3 Hz, 1H), 6.37 (d, *J* = 7.8 Hz, 2H), 3.63 (d, *J* = 13.0 Hz, 1H), 3.56 (t, *J* = 6.2 Hz, 1H), 3.46 (d, *J* = 13.0 Hz, 1H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 167.2, 147.2, 145.2, 135.0, 132.9, 130.9, 130.1, 129.1(2), 129.0(8), 127.5, 127.2, 123.7, 122.5, 118.2, 113.4, 91.4, 49.2. HRMS (ESI) $[M+H]^+$: calculated for $C_{21}H_{19}N_2O_2$: 331.1441, found 331.1450.



3-(1-Hydroxy-3-oxo-2-phenylisoindolin-1-yl)propanenitrile (8g). The product **8g** was obtained in 35% (19.5 mg) yield as a yellow oil after column chromatography. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.65-7.62 (m, 1H), 7.55 (d, *J* = 7.5 Hz, 1H), 7.46-7.38 (m, 4H), 7.35-7.28 (m, 3H), 4.62 (br, 1H), 2.41-2.35 (m, 1H), 2.31-2.25 (m, 1H), 2.08-2.01 (m, 1H), 1.80-1.74 (m, 1H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 166.7, 144.6, 134.6, 133.4, 130.5, 130.4, 129.2, 127.3, 125.9, 124.0, 122.0, 118.1, 91.4, 31.9, 12.0. This compound has been reported in the published literature.[20]

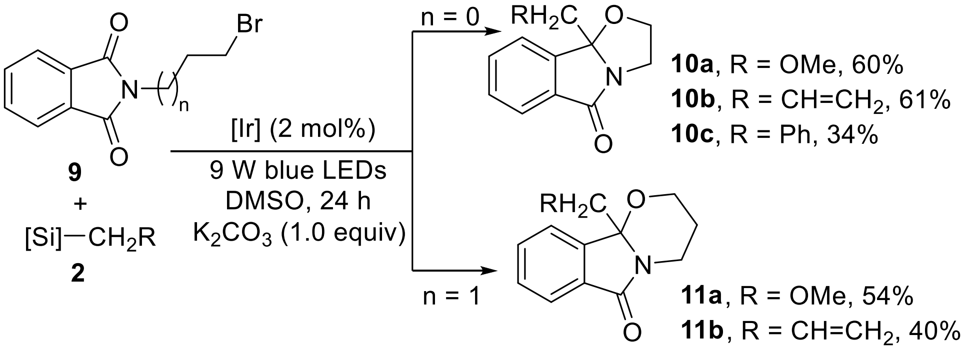


3-Allyl-3-hydroxy-2-phenylisoindolin-1-one (8h). The product **8h** was obtained in 93% (49.3 mg) yield as a yellow solid after column chromatography. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.57-7.52 (m, 2H), 7.46-7.43 (m, 2H), 7.36-7.23 (m, 5H), 5.24-5.15 (m, 1H), 4.84 (d, *J* = 10.1 Hz, 1H), 4.75-4.71 (m, 1H), 4.59 (s, 1H), 2.80-2.76 (m, 1H), 2.63-2.59 (m, 1H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 167.0, 146.2, 135.3, 132.5, 130.6, 130.5, 129.5, 128.6, 126.8, 126.6, 123.4, 122.1, 119.8, 92.2, 40.6. HRMS (ESI) $[M+H]^+$: calculated for $C_{17}H_{16}NO_2$: 266.1176, found 266.1183.

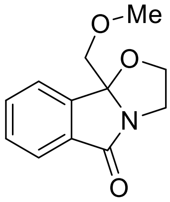


3-Benzyl-3-hydroxy-2-phenylisoindolin-1-one (8i). The product **8i** was obtained in 40% (25.2 mg) yield as a white solid after column chromatography. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.57-7.55 (m, 2H), 7.48-7.44 (m, 1H), 7.38-7.27 (m, 5H), 7.16 (d, *J* = 7.6 Hz, 1H), 7.11-7.08 (m, 1H), 7.04-7.01 (m, 2H), 6.66 (d, *J* = 7.3 Hz, 2H), 4.17 (s, 1H), 3.42 (d, *J* = 14.1 Hz, 1H), 3.06 (d,*J* = 14.1 Hz, 1H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 166.7, 145.9, 135.8, 134.2, 132.2, 130.5, 130.4, 129.6, 128.6, 127.7, 126.9, 126.5, 126.0, 123.5, 122.9, 92.9, 42.8. This compound has been reported in the published literature.[21]

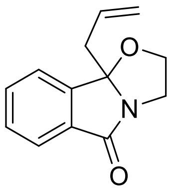
3 General procedure of radical-radical cross-coupling reaction/anionic cyclisation cascade process.



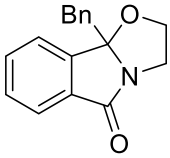
To an oven dried transparent 10 mL Schlenk tube equipped with stirring bar, Ir[dF(CF$_3$)ppy)]$_2$(dtbbpy)PF$_6$ (4.5 mg, 0.004 mmol, 2 mol %), K$_2$CO$_3$ (27.6 mg, 0.2 mmol, 1.0 equiv), and *N*-(ω-bromoalkyl)phthalimide **9** (0.2 mmol, 1.0 equiv) were added. Then, potassium [18-Crown-6] bis(catecholato)-alkylsilicate (0.4 mmol, 2.0 equiv) were added intothe tube. The tube was sealed with a rubber septum.The tube was then charged with degassed DMSO (6.0 mL, 0.033 M) via a syringe under N$_2$. The tube was irradiated with a 9 W blue LEDs strip spiraled within a bowel for 24 h (cooling with a fan). After the reaction was complete, the reaction solution was diluted with saturated Na$_2$CO$_3$ aqueous solution, and was extracted with EtOAc (4 x 5 mL). The organic layer was washed with brine, dried over MgSO$_4$, filtered, and solvent was evaporated to obtain crude product. Flash chromatography over silica gel afforded the product.



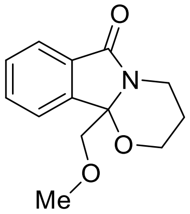
9B-(methoxymethyl)-2,3-dihydrooxazolo[2,3-*a*]isoindol-5(9b*H*)-one (10a). The product **10a** was obtained in 60% (26.3 mg) yield as a yellow oil after column chromatography. ^{1}H NMR (500 MHz, CDCl$_3$) δ 7.77 (d, *J* = 7.5 Hz, 1H), 7.62-7.57 (m, 2H), 7.54-7.51 (m, 1H), 4.33-4.30 (m, 1H), 4.17-4.13 (m, 1H), 4.06 (q, *J* = 7.7 Hz, 1H), 3.78 (d, *J* = 10.5 Hz, 1H), 3.73 (d, *J* = 10.5 Hz, 1H), 3.52-3.47 (m, 1H), 3.43 (s, 3H). ^{13}C NMR (126 MHz, CDCl$_3$) δ 173.8, 144.9, 133.1, 132.2, 130.4, 124.3, 123.1, 98.6, 74.8, 70.3, 59.9, 43.2. HRMS (ESI) [M+H]$^+$: calculated for C$_{12}$H$_{14}$NO$_3$: 220.0974, found 220.0971.



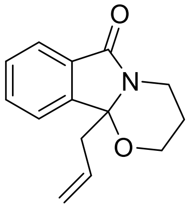
9B-allyl-2,3-dihydrooxazolo[2,3-*a*]isoindol-5(9b*H*)-one (10b). The product **10b** was obtained in 61% (26.3 mg) yield as a yellow liquid after column chromatography. ^{1}H NMR (500 MHz, CDCl$_3$) δ 7.76-7.74 (m, 1H), 7.60-7.54 (m, 2H), 7.52-7.48 (m, 1H), 5.70-5.61 (m, 1H), 5.13-5.09 (m, 1H), 5.06-5.04 (m, 1H), 4.26-4.23 (m, 1H), 4.18-4.14 (m, 1H), 4.05 (q, *J* = 7.8 Hz, 1H), 3.46-3.40 (m, 1H), 2.78 (d, *J* = 7.1 Hz, 2H). ^{13}C NMR (126 MHz, CDCl$_3$) δ 173.7, 146.3, 133.0, 132.0, 131.4, 130.0, 124.1, 122.7, 119.7, 99.8, 69.6, 42.6, 40.5. HRMS (ESI) [M+H]$^+$: calculated for $C_{13}H_{14}NO_2$: 216.1025, found 216.1019.



9B-benzyl-2,3-dihydrooxazolo[2,3-*a*]isoindol-5(9b*H*)-one (10c). The product **10c** was obtained in 34% (18.2 mg) yield as a yellow oil after column chromatography. ^{1}H NMR (500 MHz, CDCl$_3$) δ 7.71 (d, *J* = 7.5 Hz, 1H), 7.60-7.57 (m, 1H), 7.51-7.48 (m, 2H), 7.26-7.22 (m, 5H), 4.03-3.97 (m, 2H), 3.94-3.90 (m, 1H), 3.39 (d, *J* = 13.8 Hz, 1H), 3.16 (d, *J* = 13.9 Hz, 1H), 2.84-2.78 (m, 1H). ^{13}C NMR (126 MHz, CDCl$_3$) δ 173.9, 147.1, 135.2, 133.0, 131.8, 130.6, 130.0, 128.1, 127.0, 124.2, 122.6, 100.5, 69.8, 43.2, 43.1. This compound has been reported in the published literature.[22]

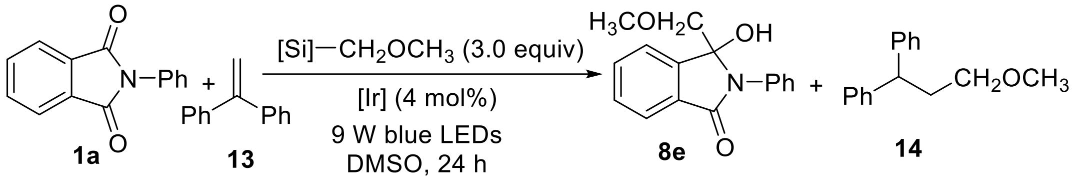


10B-(methoxymethyl)-3,4-dihydro-2*H*-[1,3]oxazino[2,3-a]isoindol-6(10b*H*)-one (11a). The product **11a** was obtained in 54% (25.1 mg) yield as a yellow oil after column chromatography. ^{1}H NMR (500 MHz, CDCl$_3$) δ 7.83 (d, *J* = 7.4 Hz, 1H), 7.67 (d, *J* = 7.4 Hz, 1H), 7.58-7.55 (m, 1H), 7.53-7.50 (m, 1H), 4.48-4.44 (m, 1H), 4.25-4.20 (m, 1H), 4.17 (d, *J* = 10.5 Hz, 1H), 4.00-3.96 (m, 1H), 3.59 (d, *J* = 10.5 Hz, 1H), 3.41 (s, 3H), 3.33-3.27 (m, 1H), 1.89-1.79 (m, 1H), 1.66-1.62 (m, 1H). ^{13}C NMR (126 MHz, CDCl$_3$) δ 167.3, 145.5, 132.1, 131.5, 129.8, 123.7, 122.7, 87.9, 70.5, 62.3, 59.7, 35.9, 24.7. HRMS (ESI) [M+H]$^+$: calculated for $C_{20}H_{17}N_2O_2$: 317.1290, found 317.1284.

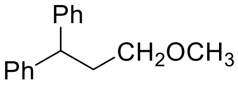


10B-allyl-3,4-dihydro-2*H*-[1,3]oxazino[2,3-a]isoindol-6(10b*H*)-one (11b). The product **11b** was obtained in 40% (18.1 mg) yield as a yellow oil after column chromatography. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.81 (d, *J* = 7.5 Hz, 1H), 7.58-7.48 (m, 3H), 5.41-5.33 (m, 1H), 5.05-4.98 (m, 2H), 4.46-4.42 (m, 1H), 4.23-4.17 (m, 1H), 3.97-3.93 (m, 1H), 3.27-3.21 (m, 1H), 3.04-2.95 (m, 2H), 1.87-1.77 (m, 1H), 1.64-1.61 (m, 1H). ^{13}C NMR (126 MHz, CDCl3) δ 166.7, 145.7, 132.0, 131.8, 130.4, 129.6, 123.7, 121.9, 119.6, 89.4, 61.6, 35.3, 35.0, 24.9. HRMS (ESI) $[M+Na]^+$: calculated for $C_{20}H_{17}NO_3Na$: 342.1106, found 342.1101.

4 Experiment dealing with interception of radical and proposed mechanism

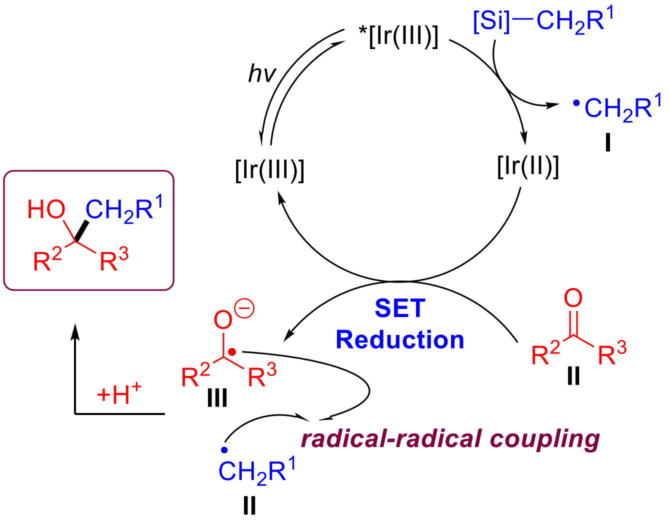


To an oven dried transparent 10 mL Schlenk tube equipped with stirring bar, $Ir[dF(CF_3)ppy)]_2(dtbbpy)PF_6$ (4.5 mg, 0.004 mmol, 4 mol %), *N*-phenylphthalimide (22.3 mg, 0.1mmol, 1.0 equiv), and 1,1-diphenylethylene **13** (18.0 mg, 0.1 mmol, 1.0 equiv) were added. Then, potassium [18-Crown-6] bis(catecholato)-methoxymethylsilicate (177.8 mg, 0.3 mmol, 3.0 equiv) were added in the tube. The tube was sealed with a rubber septum. The tube was then charged with degassed DMSO (6.0 mL, 0.033 M) via a syringe under N_2. The tube was irradiated with a 9 W blue LEDs strip spiraled within a bowel for 24 h (cooling with a fan). After the reaction was complete, the reaction solution was diluted with saturated Na_2CO_3 aqueous solution, and was extracted with EtOAc (4 x 5 mL). The organic layer was washed with brine, dried over $MgSO_4$, filtered, and solvent was evaporated to obtain crude product. Flash chromatography over silica gel afforded the product **8e** and **14**.



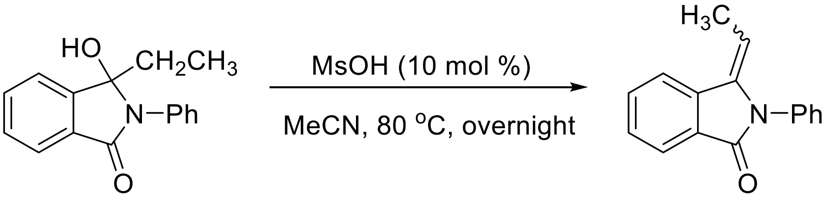
(3-Methoxypropane-1,1-diyl)dibenzene (16). The product **16** was obtained in 50% (22.6 mg) yield as a yellow oil after column chromatography. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.32-7.26(m, 8H), 7.21-7.18 (m, 2H), 4.14 (t, *J* = 7.9 Hz, 1H), 3.34-3.31 (m, 5H), 2.37-2.32 (m, 2H).^{13}C NMR (126 MHz, $CDCl_3$) δ 144.6, 128.4, 127.9, 126.1, 70.6, 58.5, 47.2, 35.3. This compound has been reported in the published literature.[23]

Based on the above results and related literature reports, we proposed the mechanism depicted in Scheme SI-1. The initial SET between the photoexcited photocatalyst and bis-catecholato alkylsilicate generates primary alkyl radical **I**. After an SET reduction of carbonyl group of **II** ($E_{1/2,\ red}$ = -1.37 V vs SCE) by the reduced photocatalyst ($E_{1/2}(Ir^{II}/Ir^{III})$ = -1.31 V vs SCE for **1a**), a ketyl radical **III** was formed and the photocatalyst was regenerated in its ground state, thus closing the redox-neutral photocatalytic cycle. Lastly, radical-radical coupling of ketyl radical **III** with primary alkyl radical **II** produces the expected alcohol product with protonation.



Scheme SI-1 Proposed mechanism.

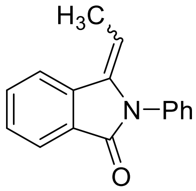
5 Methanesulfonic acid-catalysed dehydration reaction



3a

12, 74%, *E*/*Z*=70:30

To an oven dried transparent 10 mL Schlenk tube equipped with stirring bar, 3-ethyl-2,3-dihydro-3-hydroxy-2-phenyl-1*H*-isoindol-1-one **3a** (51.0 mg, 0.2 mmol, 1.0 equiv), MeCN (1.0 mL, 0.2 M) and methanesulfonic acid (1.9 mg, 0.02 mmol, 10 mol %) were added in the tube. The resulting mixture was stirred at 80 °C for overnight. After the reaction was complete, the reaction solution was diluted with saturated Na_2CO_3 aqueous solution, and was extracted with EtOAc (4 x 5 mL). The organic layer was washed with brine, dried over $MgSO_4$, filtered, and solvent was evaporated to obtain crude product. Flash chromatography over silica gel afforded the product.



3-Ethylidene-2-phenylisoindolin-1-one (12). The product **12** was obtained in 74% (34.8 mg) yield as a yellow solid after column chromatography. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.99-7.89(m, 2H), 7.68-7.64 (m, 1H), 7.61-7.47 (m, 3H), 7.43-7.40 (m, 1H), 7.38-7.32 (m, 2H),[5.77-5.73 (m, 0.28H), 5.41-5.37 (m, 0.72H)], [2.17 (d, J = 7.7 Hz, 2.16H), 1.38 (d, J = 7.7Hz, 0.84H)]. ^{13}C NMR (126 MHz, $CDCl_3$) δ 167.9, 166.0, 138.0, 137.7, 137.1, 135.5, 134.9,132.0, 131.9(8), 130.1, 129.3, 129.1, 128.9, 128.8, 128.6, 128.4, 128.1, 127.8, 123.8, 123.5,123.3, 118.8, 107.7, 104.0, 12.9, 12.5.This compound has been reported in the published literature.[6]

6 References

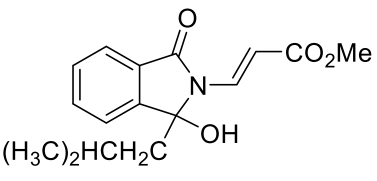
1. L. Huai, L. Zhang, Z. Wang, H. Wu and Y. Fang, *Org. Chem. Front.*, 2023, **10**, 1245−1251.
2. (*a*) G. N. Ding, B. Lu, Y. Y. Li, J. Wan, Z. G. Zhang and X. M. Xie, *Adv. Synth. Catal.*, 2015, **357**, 1013-1021; (*b*) C. A. Goodman, C. G. Hamaker and S. R. Hitchcock, *Tetrahedron Lett.*, 2013, **54**, 6012-6014; (*c*) N. H. Lu, L. H. Wang, Z. S. Li and W. Zhang, *Beilstein J. Org. Chem.*, 2012, **8**, 192-200; (*d*) Y. Bai, L. Shi, L. Zheng, S. Ning, X. Che, Z. Zhang and J. Xiang, *Org. Lett.*, 2021, **23**, 2298-2302.
3. K. Nozawa-Kumada, Y. Matsuzawa, K. Ono, M. Shigeno and Y. Kondo, *Chem. Commun.*, 2021, **57**, 8604-8607.
4. M. Kuhara and S. Komatsu, *Mem. Coll. Sci. Eng., Kyoto Imp. Univ.*, 1911, **4**, 365.
5. J. M. Dennis, C. M. Calyore, J. S. Sjoholm, J. P. Lutz, J. J. Gair and J. B. Johnson, *Synlett*, 2013, **24**, 2567-2570.
6. N. Topolovcan, F. Dupli and M. Gredicak, *Eur. J. Org. Chem.*, 2021, 3920-3924.
7. K. S. DeGlopper, J. M. Dennis and J. B. Johnson, *Tetrahedron Lett.*, 2014, **55**, 1843-1845.
8. M. Oelgemoller, P. Cygon, J. Lex and A. G. Griesbeck, *Heterocycles*, 2003, **59**, 669-684.
9. R. A. Aitken, A. D. Harper, R. A. Inwood and A. M. Z. Slawin, *J. Org. Chem.*, 2022, **87**, 4692-4701.
10. V. C. Jayawardena, K. E. Fairfull-Smith and S. E. Bottle, *Aust. J. Chem.*, 2013, **6**

6, 619-625.

11. F. Hatoum, S. Gallagher and M. Oelgemoeller, *Tetrahedron Lett.*, 2009, **50**, 6593-6596.
12. S. Gallagher, F. Hatoum, N. Zientek and M. Oelgemoeller, *Tetrahedron Lett.*, 2010, **51**, 3639-3641.
13. Y. Krishna, K. Shilpa and F. Tanaka, *Org. Lett.*, 2019, **21**, 8444-8448.
14. K. Yanagi and T. Nishiyama, *Chemischer Informationsdienst*, 1978, **9**.
15. A. G. Griesbeck and M. Oelgemoeller, *Synlett*, 1999, **1999**, 492-494.
16. M. Kapoor, D. Liu and M. C. Young, *J. Am. Chem. Soc.*, 2018, **140**, 6818-6822.
17. I. Gorokhovik, L. Neuville and J. P. Zhu, *Org. Lett.*, 2011, **13**, 5536-5539.
18. Y. F. Liang and N. Jiao, *Angew. Chem. Int. Edit.*, 2014, **53**, 548-552.
19. N. S. Mani, C. M. Mapes, J. J. Wu, X. H. Deng and T. K. Jones, *J. Org. Chem.*, 2006, **71**, 5039-5042.
20. C.-H. Yeh, Y.-C. Lin, S. Mannathan, K. Hung and C.-H. Cheng, *Adv. Synth. Catal.*, 2014, **356**, 831-842.
21. R. Kumar, V. K. Jain and N. Jain, *J. Org. Chem.*, 2022, **87**, 11939-11946.
22. T. Bousquet, J.-F. Fleury, A. Daïch and P. Netchitaïlo, *Tetrahedron*, 2006, **62**, 706-715.
23. P. C. Too, G. H. Chan, Y. L. Tnay, H. Hirao and S. Chiba, *Angew. Chem. Int. Ed.*, 2016, **55**, 3719-3723.

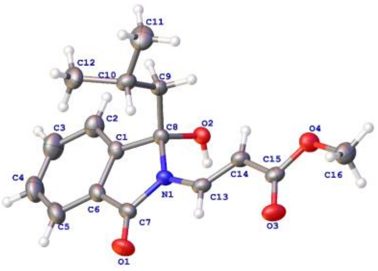
7 X-ray crystal data for compound 7b

Table 1 Crystal data and structure refinement for 7b.	
Identification code	CCDC (2284345) (**7b**)
Empirical formula	$C_{16}H_{19}NO_4$
Formula weight	289.32
Temperature/K	273.15
Crystal system	monoclinic
Space group	$P2_1/n$
a/Å	13.8240(6)
b/Å	7.5535(3)
c/Å	14.7562(6)
α/°	90
β/°	100.3540(10)
γ/°	90
Volume/Å^3	1515.75(11)
Z	4
ρ_{calc}g/cm^3	1.268
μ/mm^{-1}	0.091
F(000)	616.0
Crystal size/mm^3	0.14 × 0.11 × 0.09
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	4.458 to 50.21
Index ranges	$-16 \le h \le 16$, $-9 \le k \le 8$, $-17 \le l \le 17$
Reflections collected	32656
Independent reflections	2678 [R_{int} = 0.0415, R_{sigma} = 0.0181]
Data/restraints/parameters	2678/0/194
Goodness-of-fit on F^2	1.051
Final R indexes [I>=2σ (I)]	R_1 = 0.0400, wR_2 = 0.1099
Final R indexes [all data]	R_1 = 0.0522, wR_2 = 0.1192
Largest diff. peak/hole / e Å^{-3}	0.15/-0.15

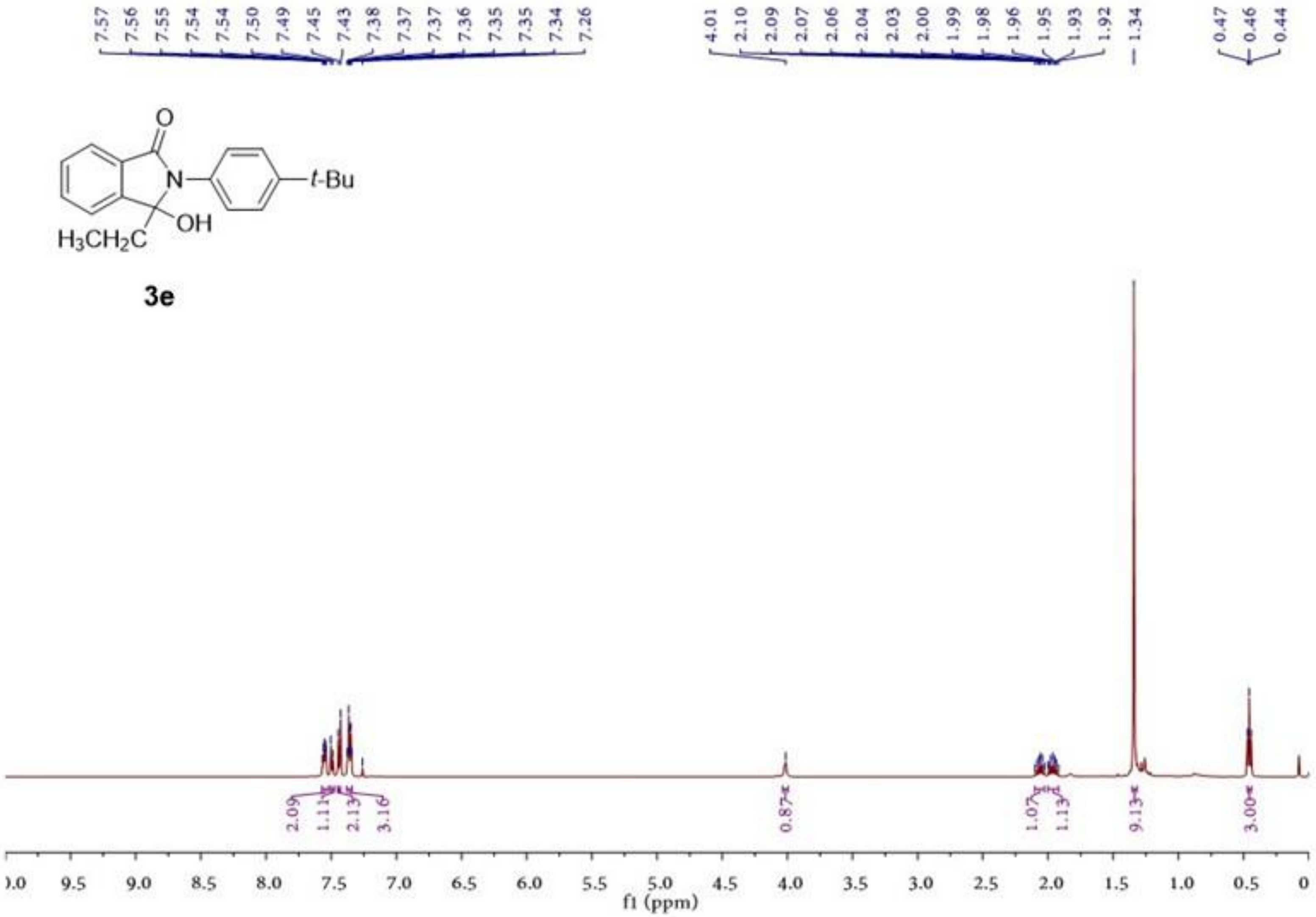


7b

$\equiv$



8 NMR spectra of new compounds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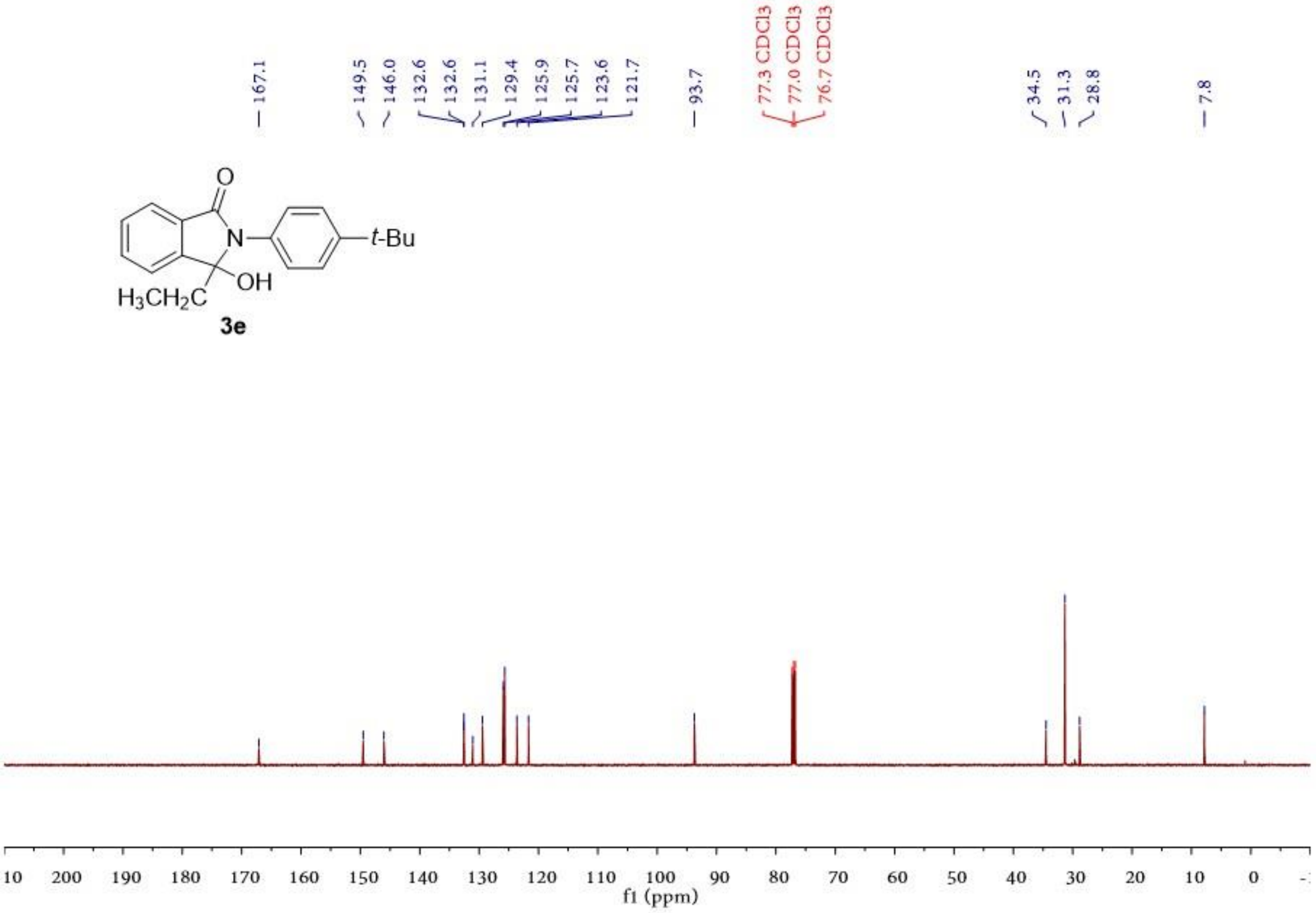
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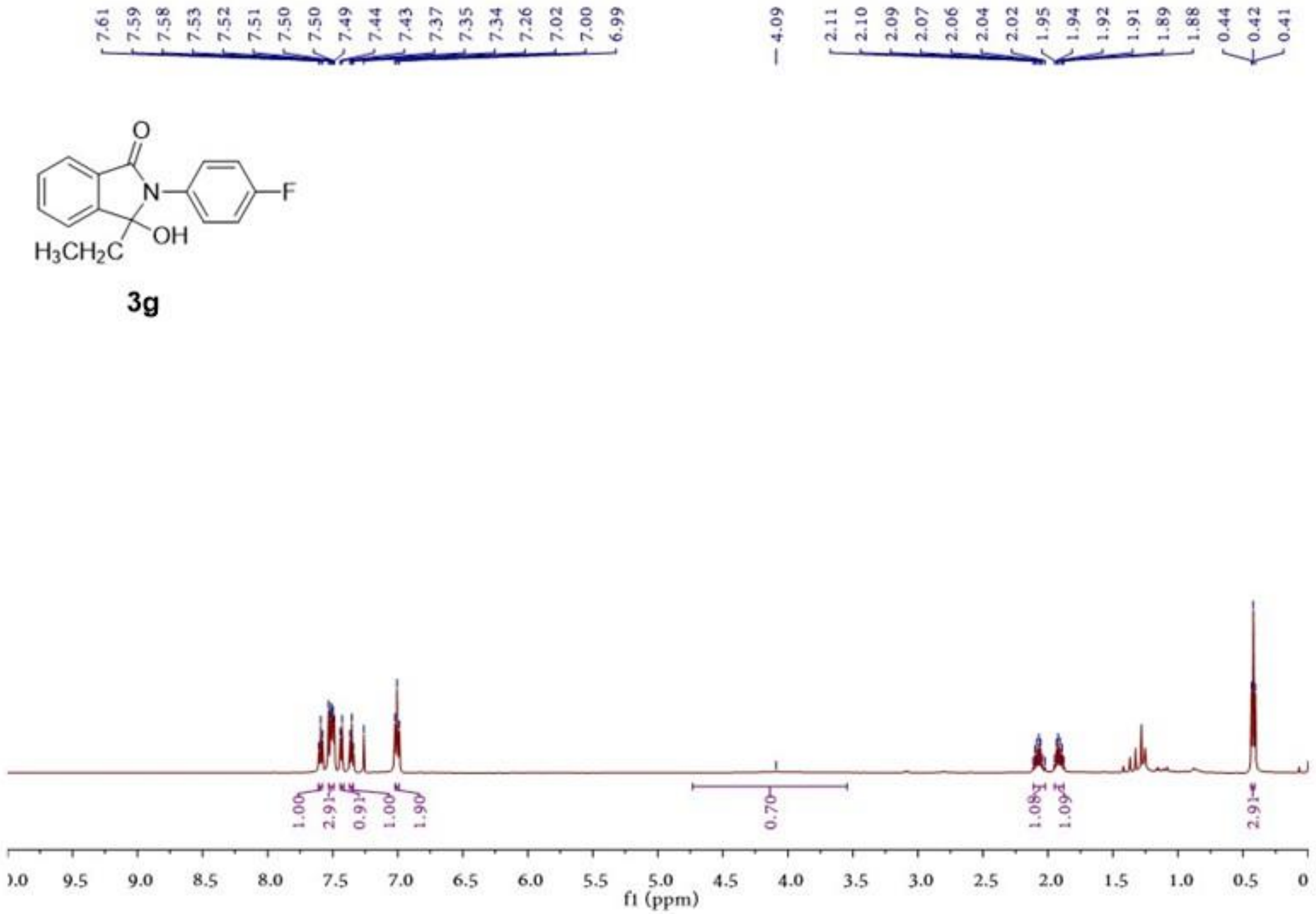
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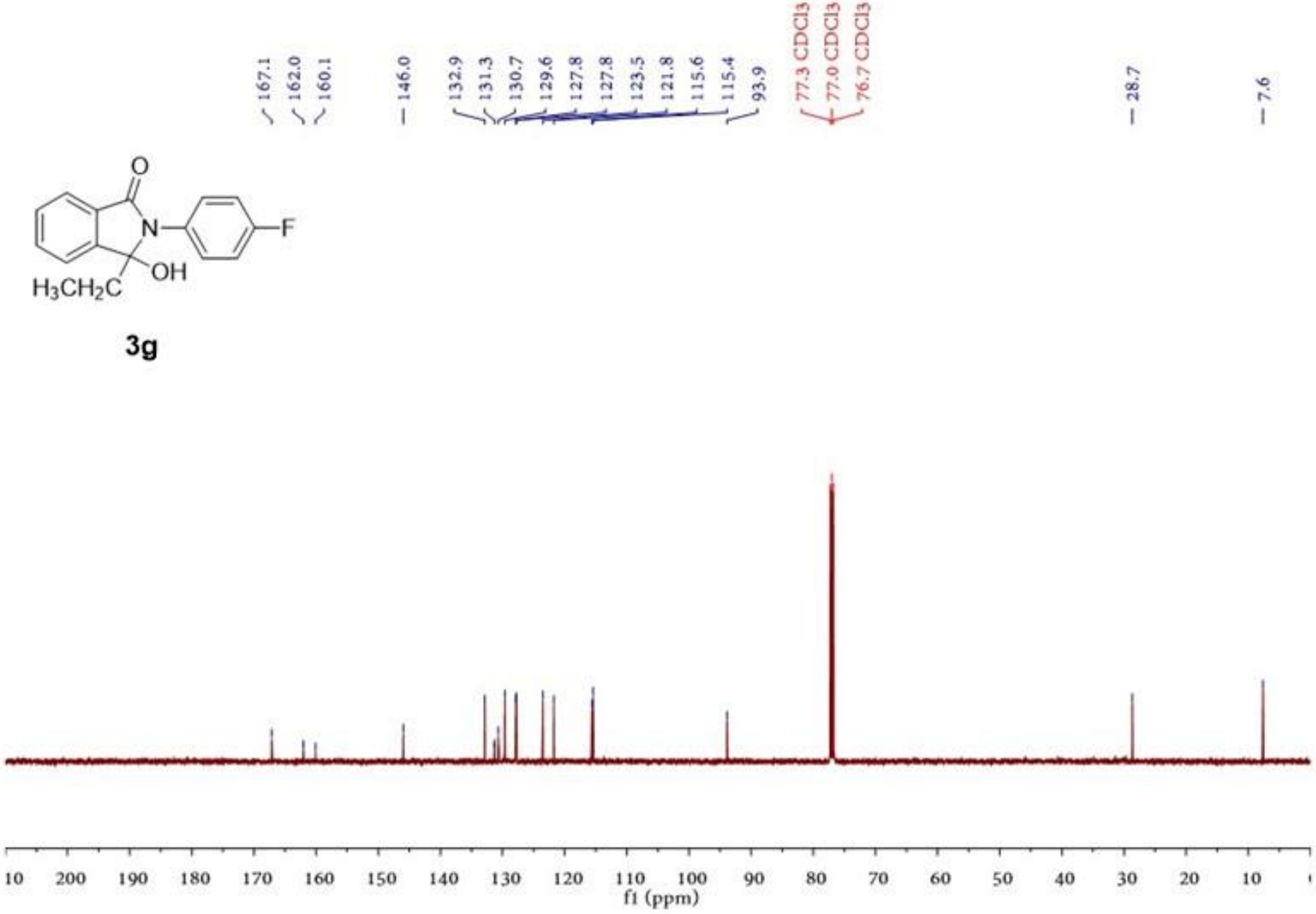
3g



3g

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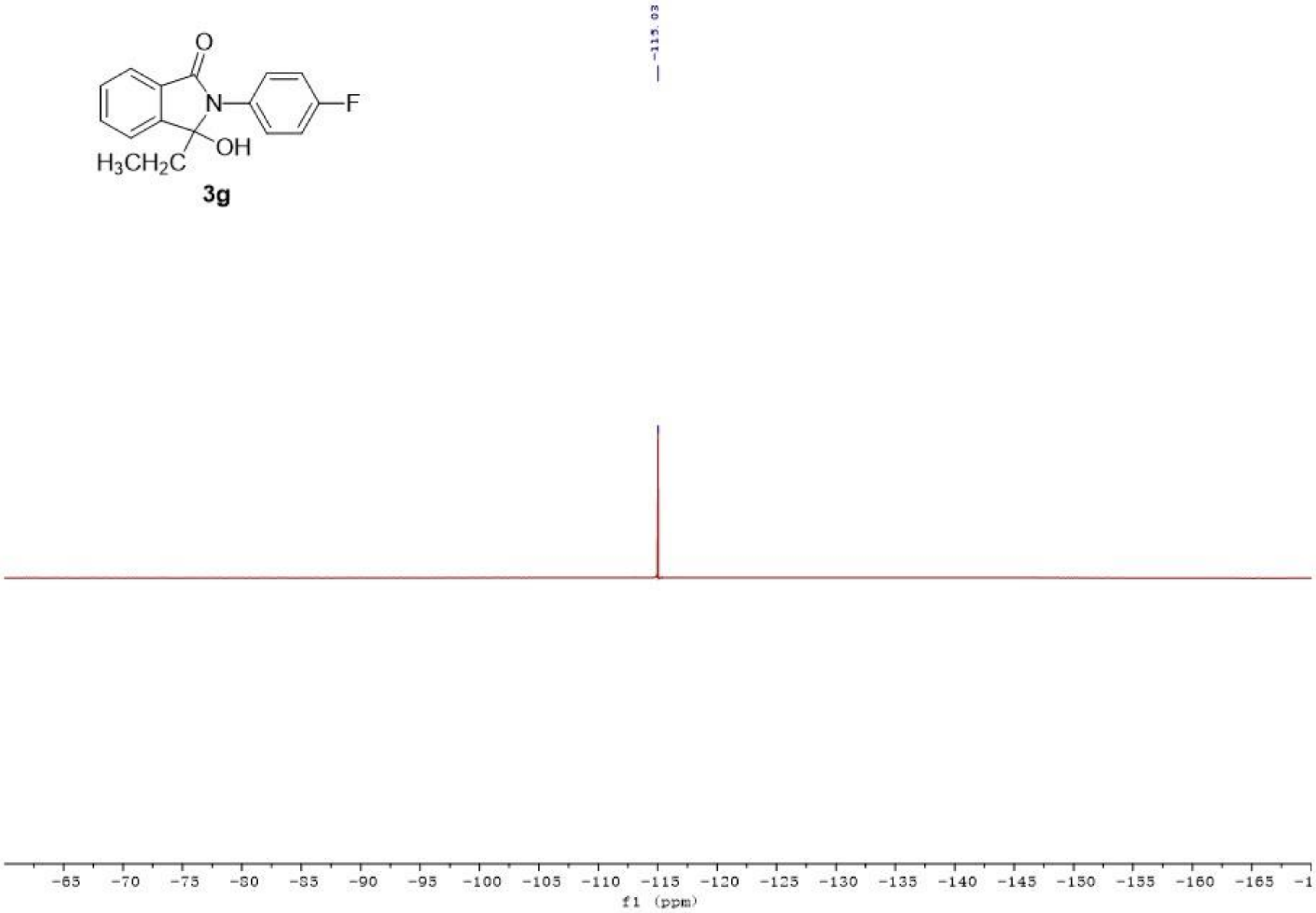
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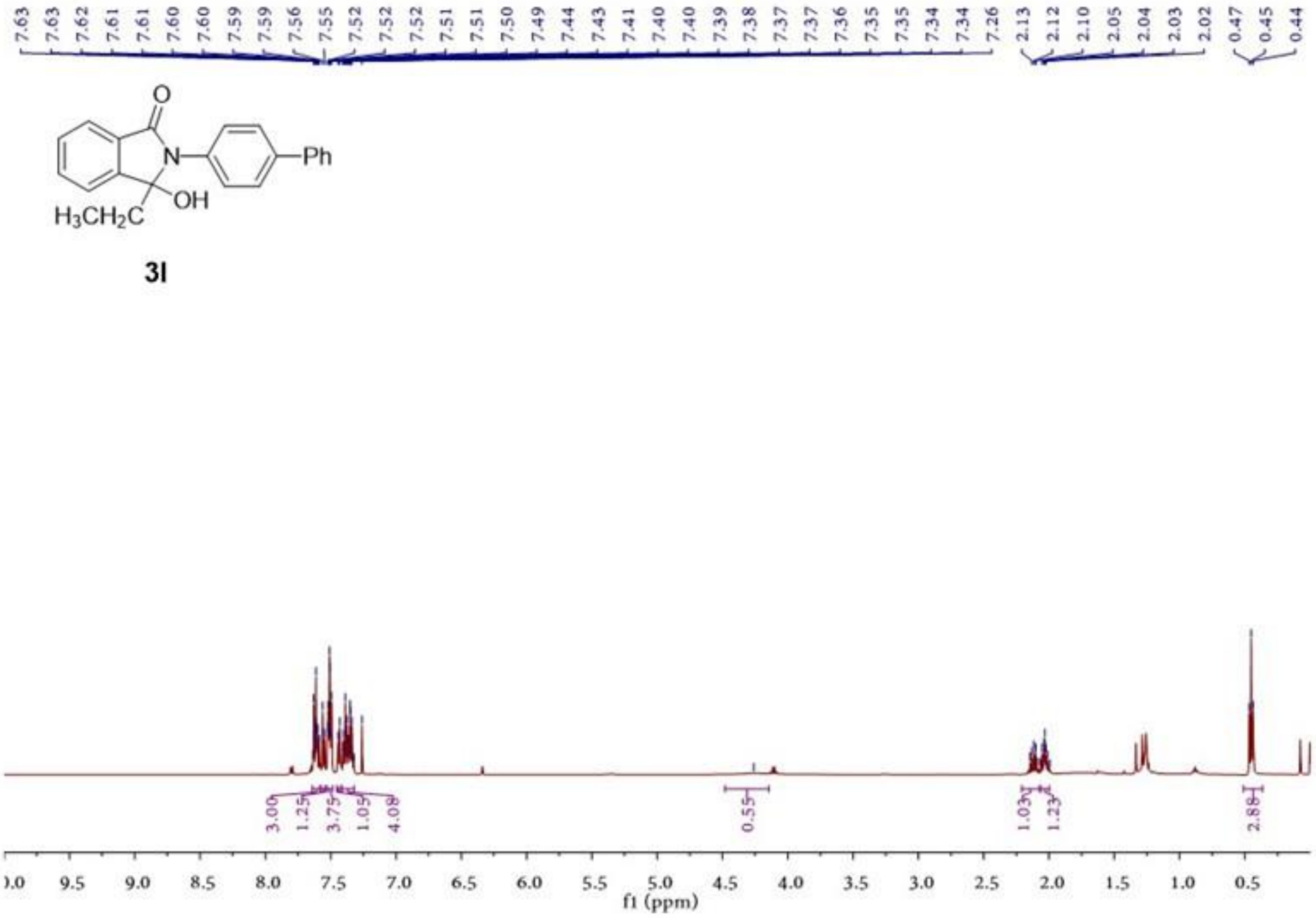
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7.34
7.34
7.26
2.13
2.12
2.10
2.05
2.04
2.03
2.02
0.47
0.45
0.44
O
N
Ph
OH
H3CH2C
3l
3.00
1.25
3.75
1.05
4.08
0.55
1.03
1.23
2.88
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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4.0
3.5
3.0
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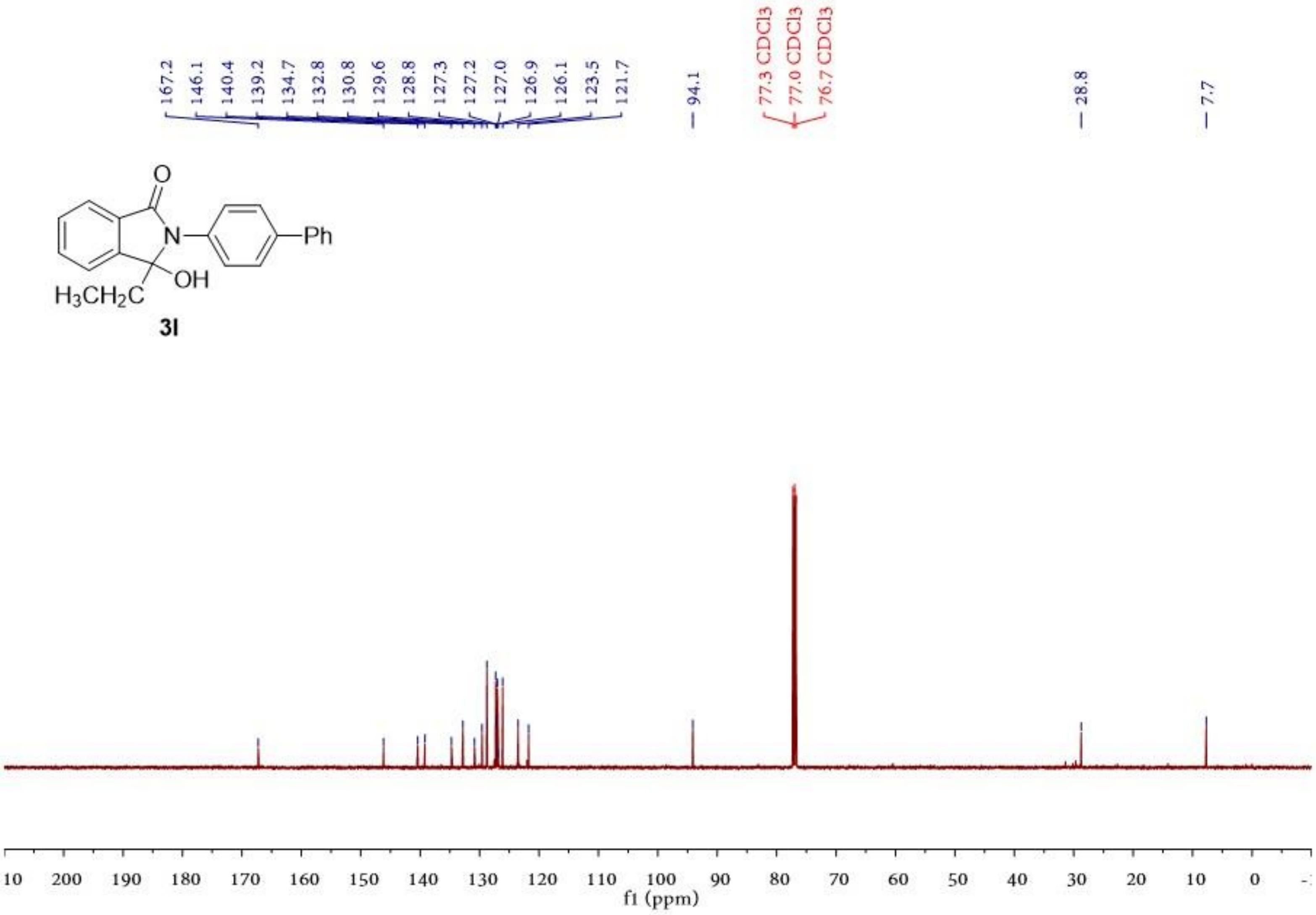
1.5
1.0
0.5
f1 (ppm)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130.8
129.6
128.8
127.3
127.2
127.0
126.9
126.1
123.5
121.7
94.1
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
28.8
7.7
O
N
Ph
OH
H_3CH_2C
3l
10
200
190
180
170
160
150
140
130
120
110
100
90
80
70
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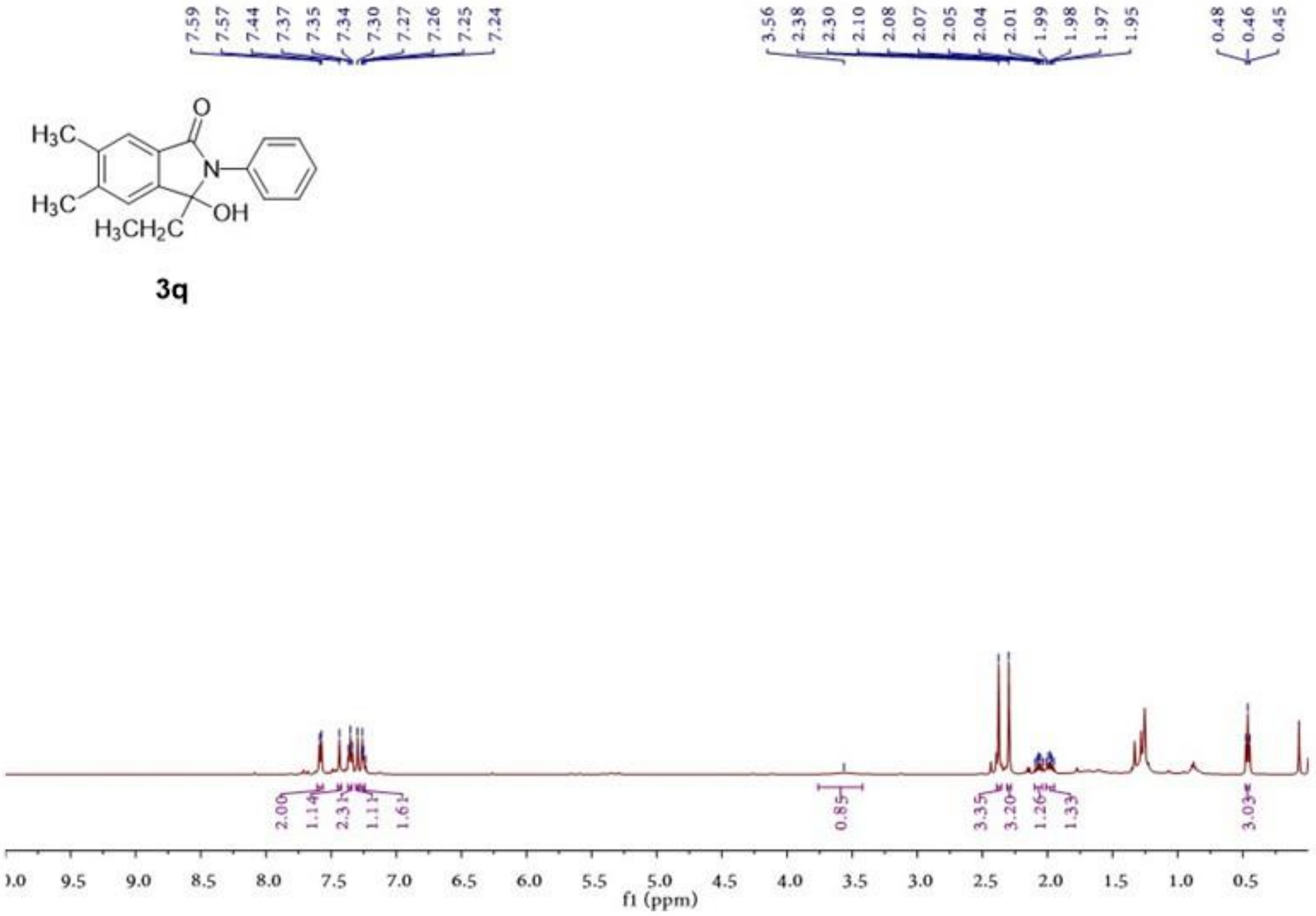
50
40
30
20
10
0
f1 (ppm)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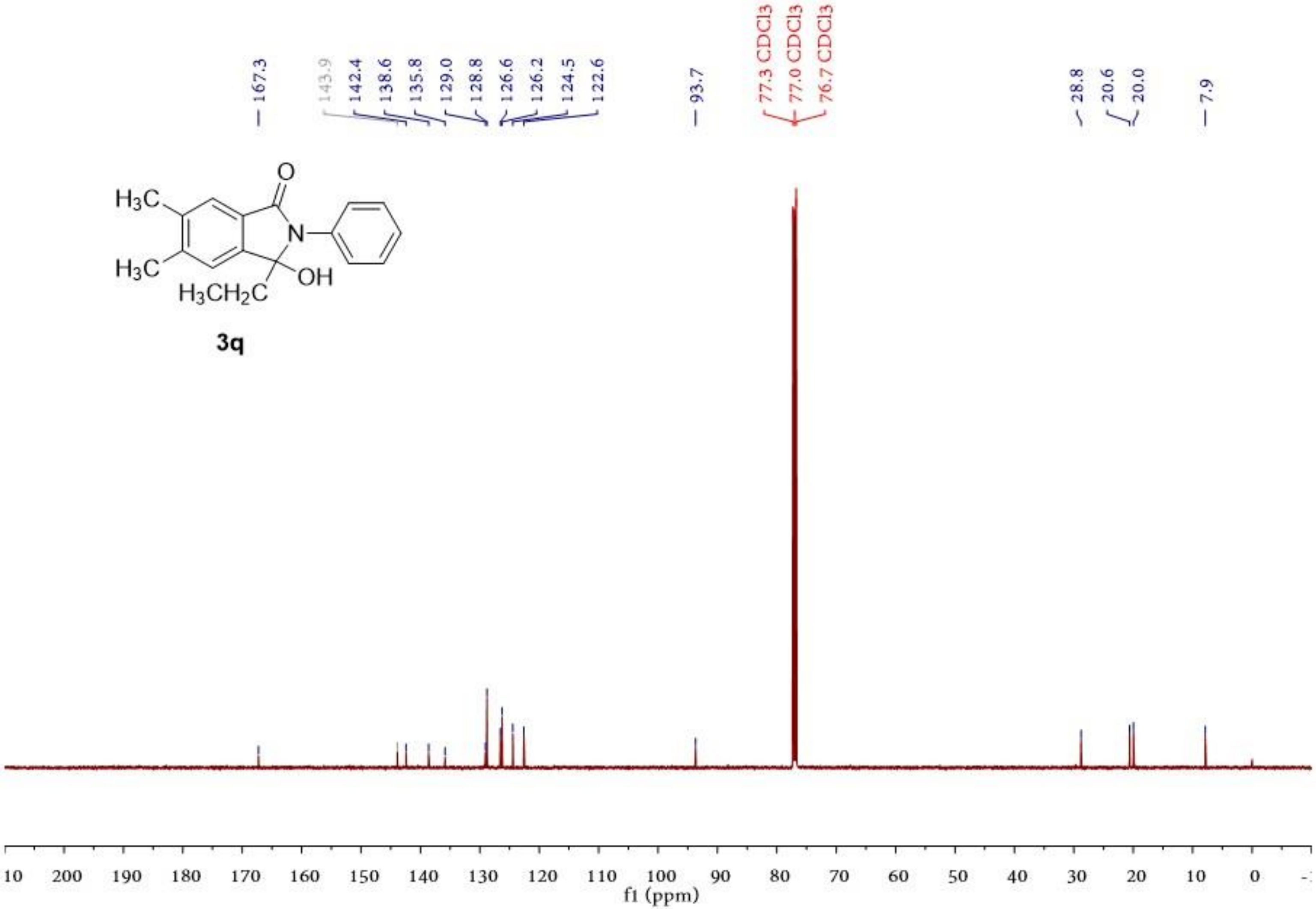
2.38
2.30
2.10
2.08
2.07
2.05
2.04
2.01
1.99
1.98
1.97
1.95
0.48
0.46
0.45
H3C
H3C
H3CH2C
O
N
OH
2.00
1.14
2.31
1.11
1.61
0.85
3.35
3.20
1.26
1.33
3.03
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

3q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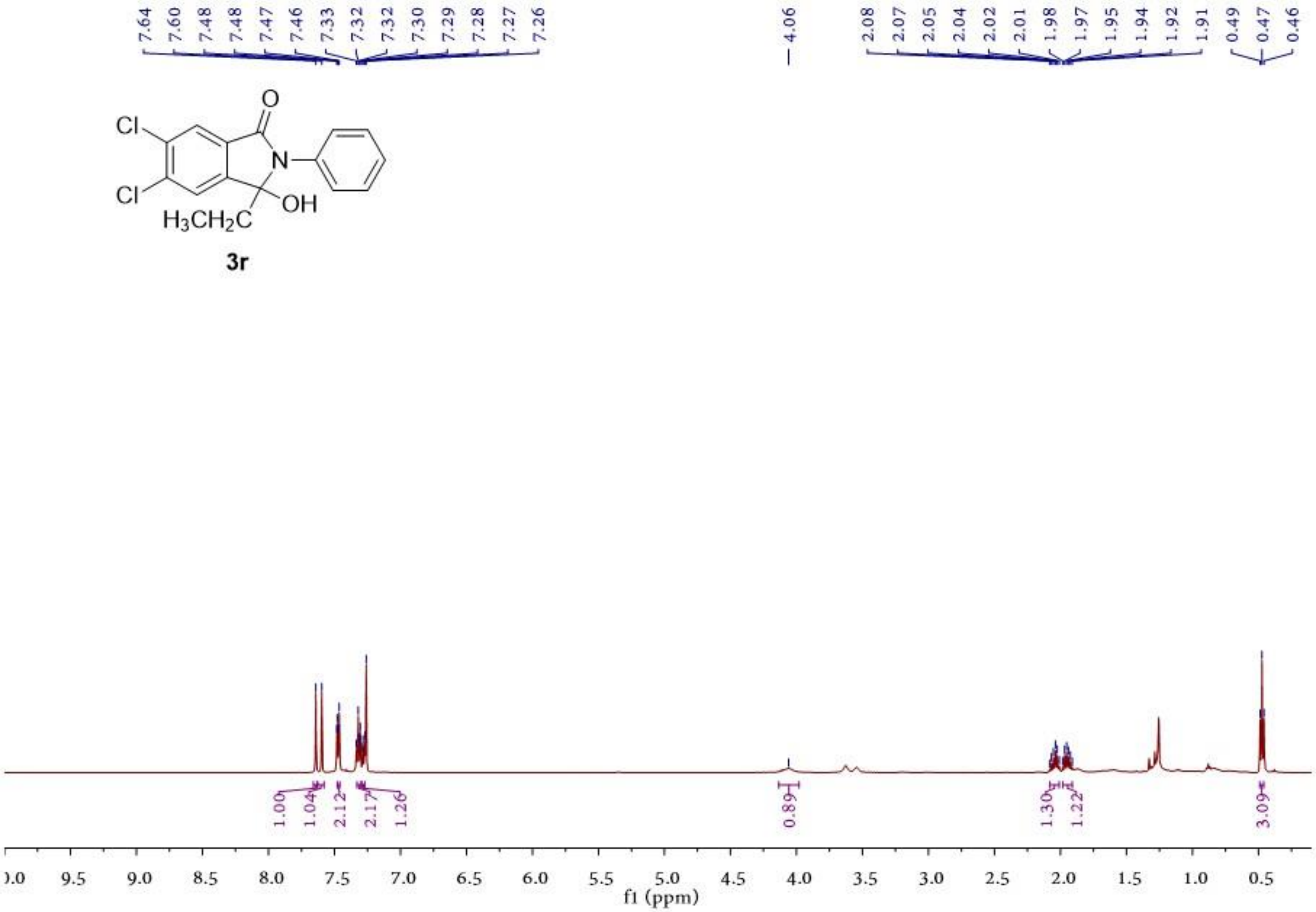
135.8
129.0
128.8
126.6
126.2
124.5
122.6
93.7
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
28.8
20.6
20.0
7.9
H_3C
H_3C
O
N
OH
H_3CH_2C
3q
10
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.30
7.29
7.28
7.27
7.26
4.06
2.08
2.07
2.05
2.04
2.02
2.01
1.98
1.97
1.95
1.94
1.92
1.91
0.49
0.47
0.46
O
Cl
N
Cl
OH
H3CH2C
3r
1.00
1.04
2.12
2.17
1.26
0.89
1.30
1.22
3.09
).0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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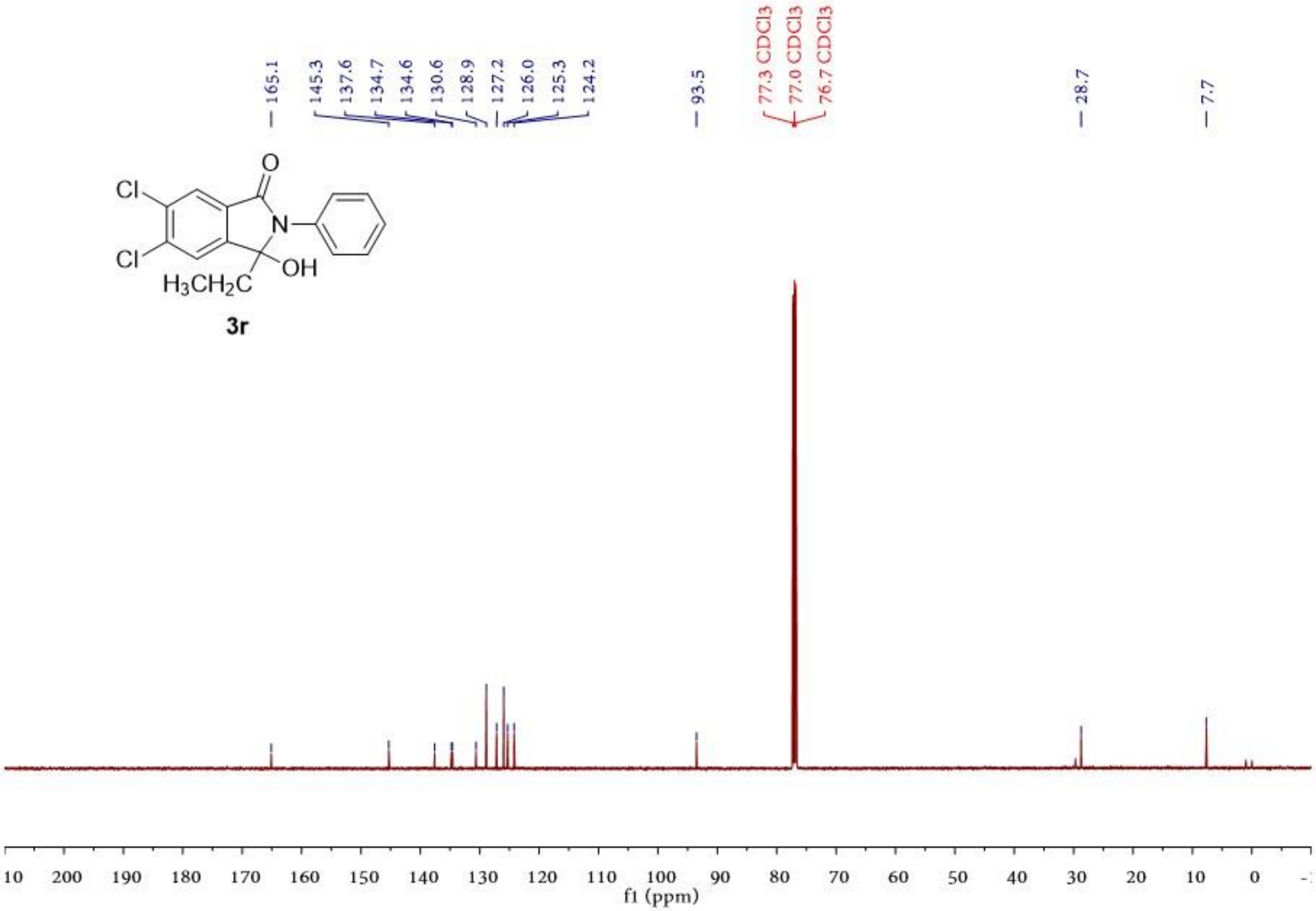
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)


3r
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134.7
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130.6
128.9
127.2
126.0
125.3
124.2
93.5
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
28.7
7.7
O
Cl
Cl
N
OH
H_3CH_2C
10
200
190
180
170
160
150
140
130
120
110
100
90
80
70
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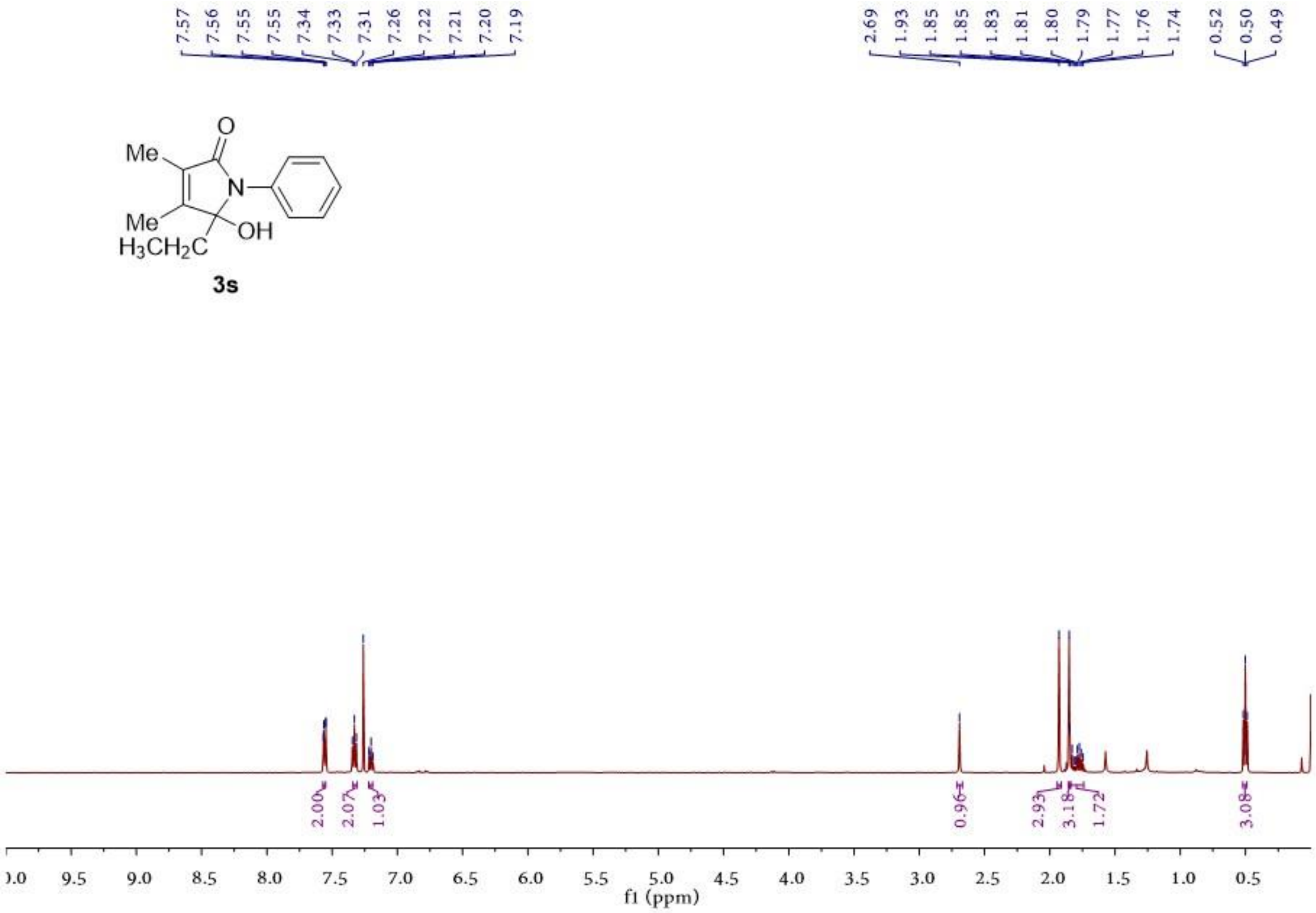
50
40
30
20
10
0
f1 (ppm)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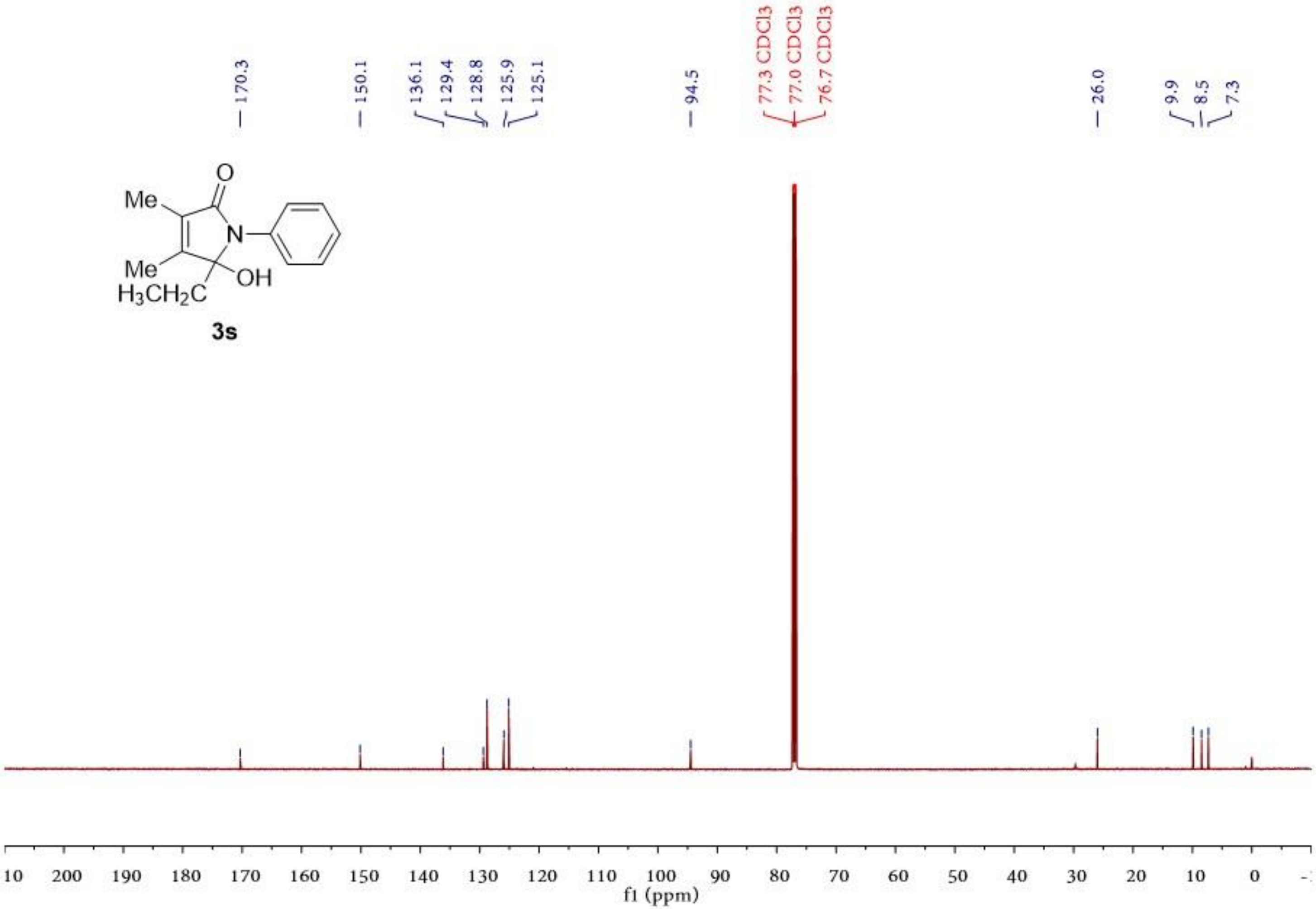
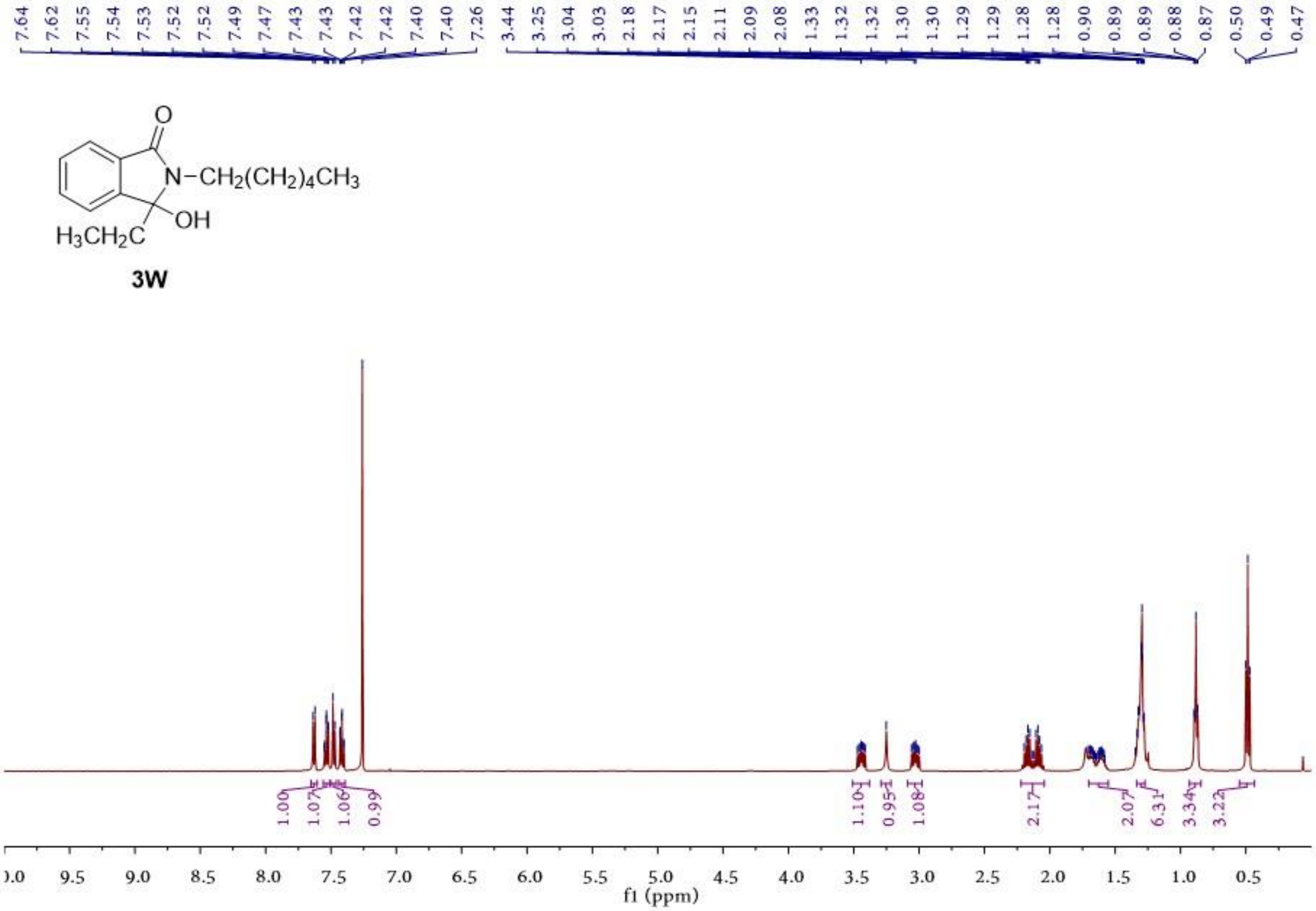
7.21
7.20
7.19
2.69
1.93
1.85
1.85
1.83
1.81
1.80
1.79
1.77
1.76
1.74
0.52
0.50
0.49
O
Me
Me
N
OH
H3CH2C
3s
2.00
2.07
1.03
0.96
2.93
3.18
1.72
3.08
).0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)


170.3
150.1
136.1
129.4
128.8
125.9
125.1
94.5
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
26.0
9.9
8.5
7.3
O
Me
N
Me
OH
H3CH2C
3s
10
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)



7.64
7.62
7.55
7.54
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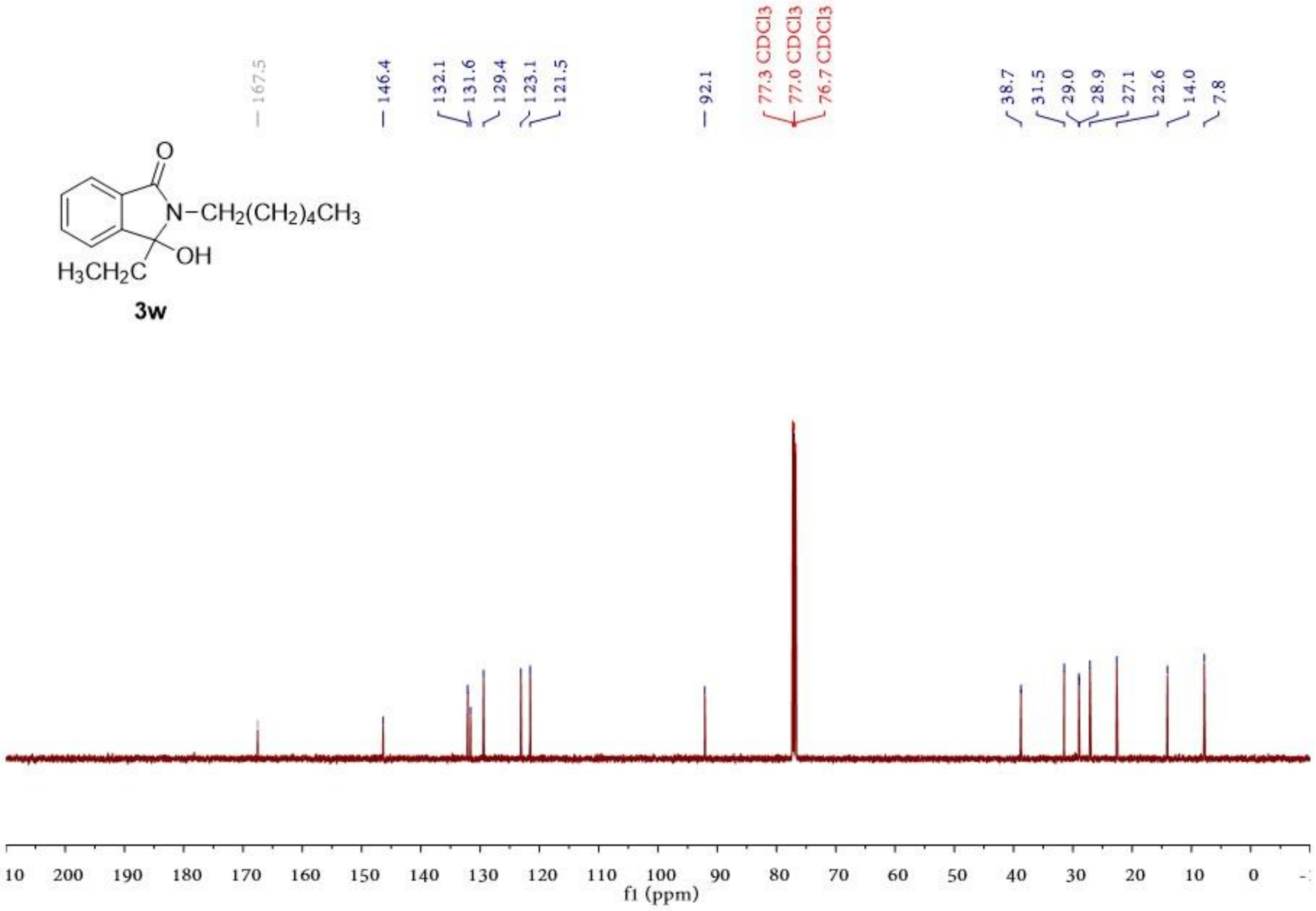
7.52
7.52
7.49
7.47
7.43
7.43
7.42
7.42
7.40
7.40
7.26
3.44
3.25
3.04
3.03
2.18
2.17
2.15
2.11
2.09
2.08
1.33
1.32
1.32
1.30
1.30
1.29
1.29
1.28
1.28
0.90
0.89
0.89
0.88
0.87
0.50
0.49
0.47
O
N−CH2(CH2)4CH3
OH
H3CH2C
1.00
1.07
1.06
0.99
1.10
0.95
1.08
2.17
2.07
6.31
3.34
3.22
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

3W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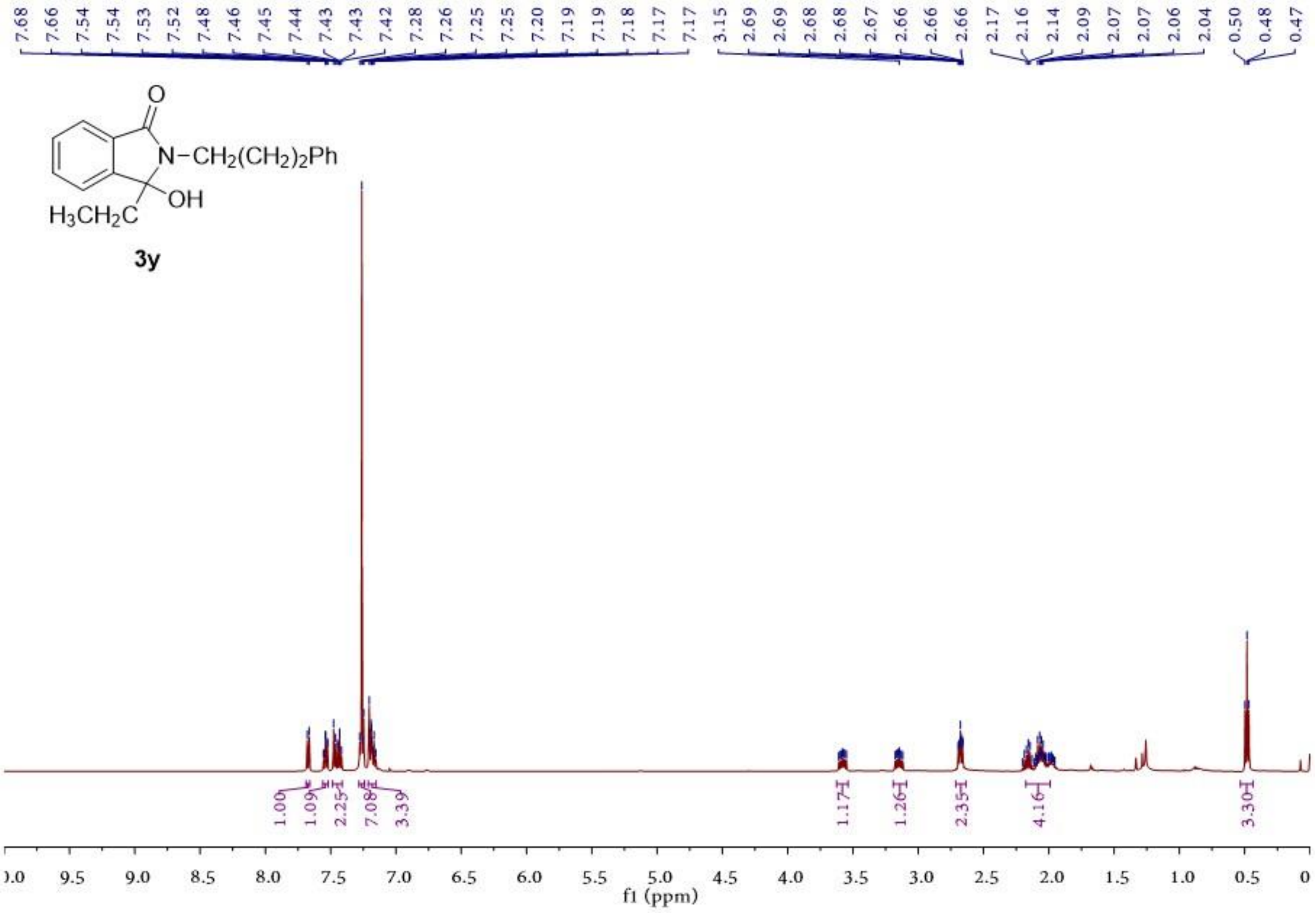
146.4
132.1
131.6
129.4
123.1
121.5
92.1
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
38.7
31.5
29.0
28.9
27.1
22.6
14.0
7.8
O
N−CH2(CH2)4CH3
OH
H3CH2C
10
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

3w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7.54
7.54
7.53
7.52
7.48
7.46
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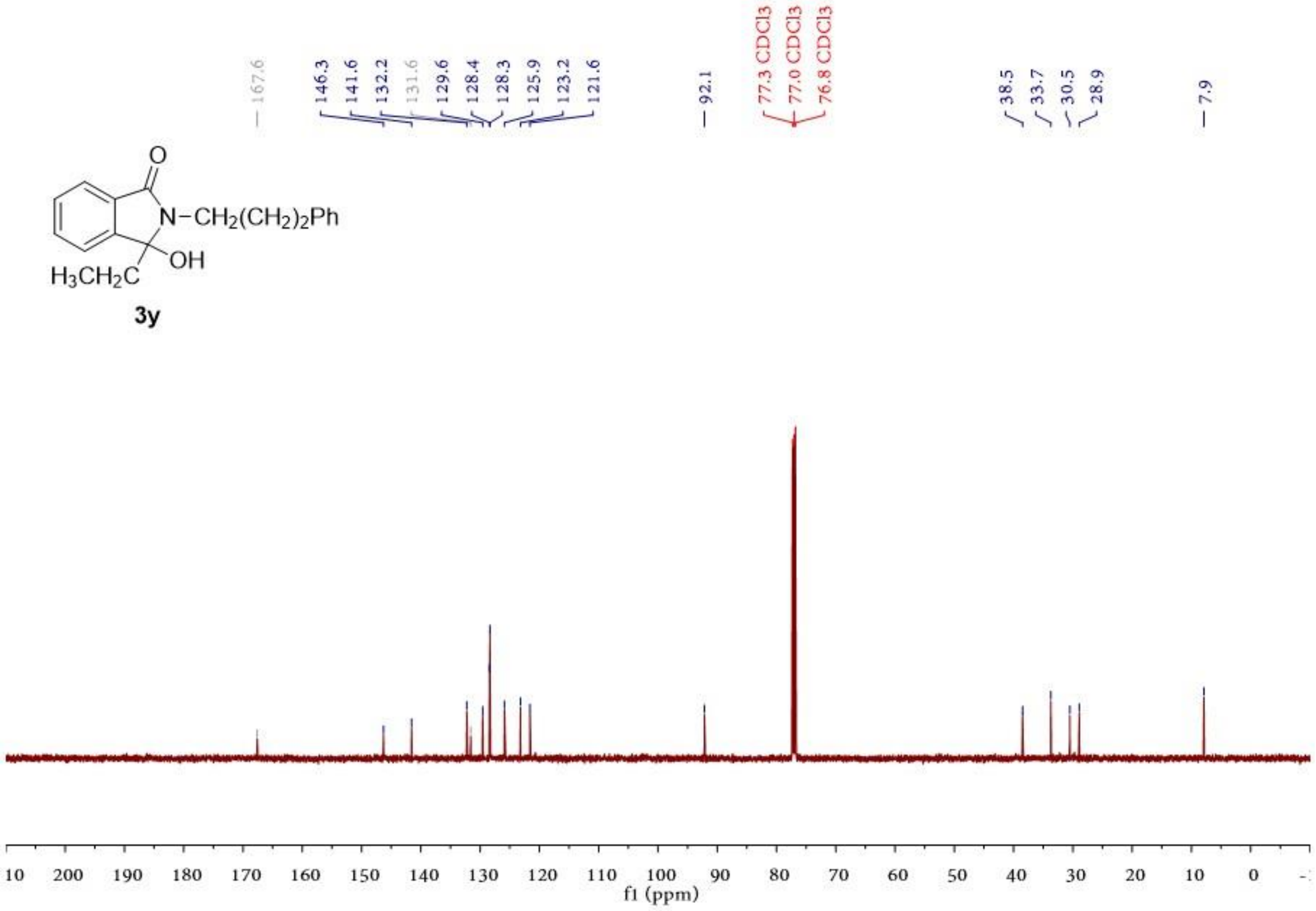
7.44
7.43
7.43
7.42
7.28
7.26
7.25
7.25
7.20
7.19
7.19
7.18
7.17
7.17
3.15
2.69
2.69
2.68
2.68
2.67
2.66
2.66
2.66
2.17
2.16
2.14
2.09
2.07
2.07
2.06
2.04
0.50
0.48
0.47
O
N−CH2(CH2)2Ph
OH
H3CH2C
1.00
1.09
2.25
7.08
3.39
1.17
1.26
2.35
4.16
3.30
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0
f1 (ppm)

3y



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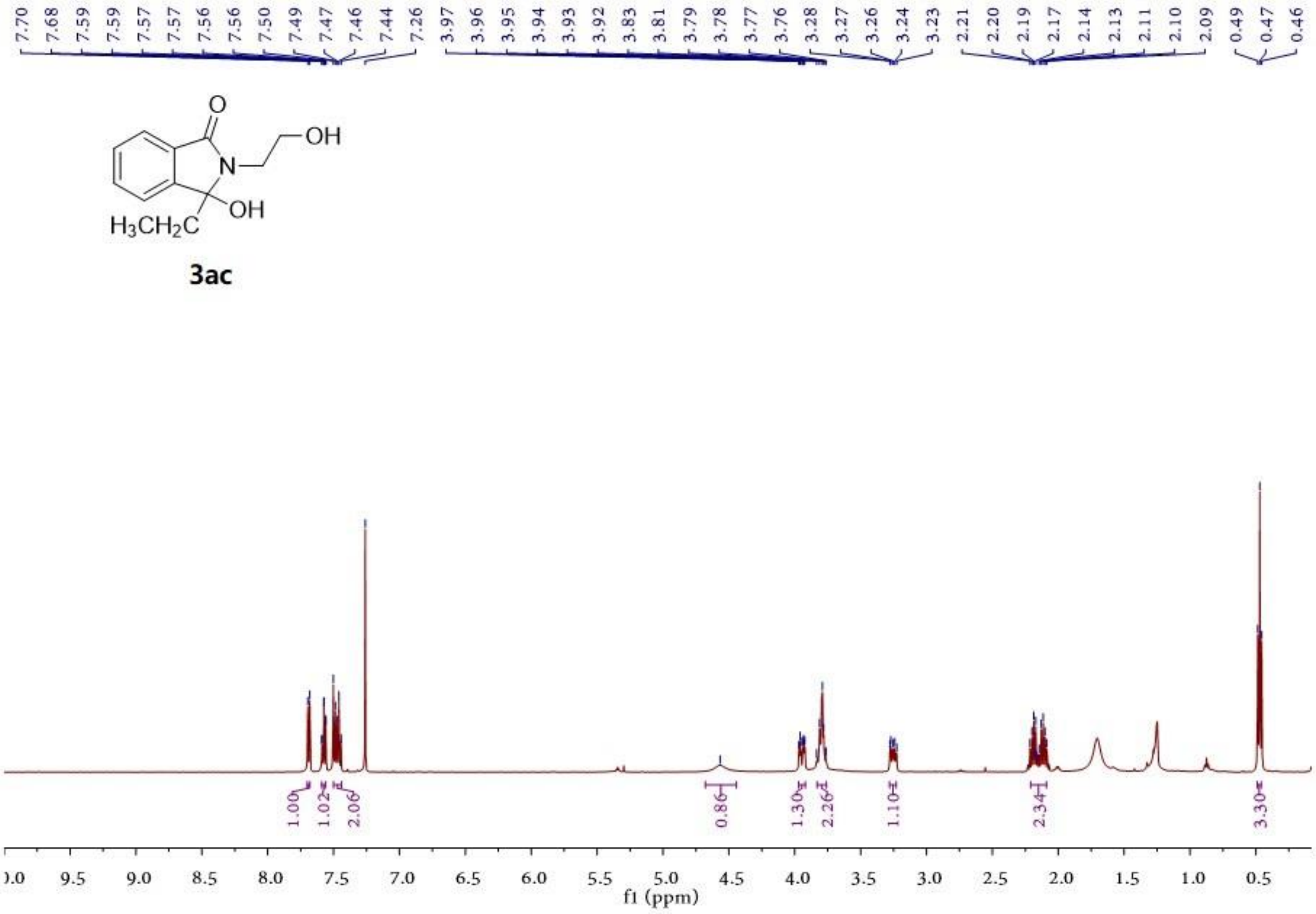
132.2
131.6
129.6
128.4
128.3
125.9
123.2
121.6
92.1
77.3 CDCl3
77.0 CDCl3
76.8 CDCl3
38.5
33.7
30.5
28.9
7.9
O
N−CH2(CH2)2Ph
OH
H3CH2C
10
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

3y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7.59
7.59
7.57
7.57
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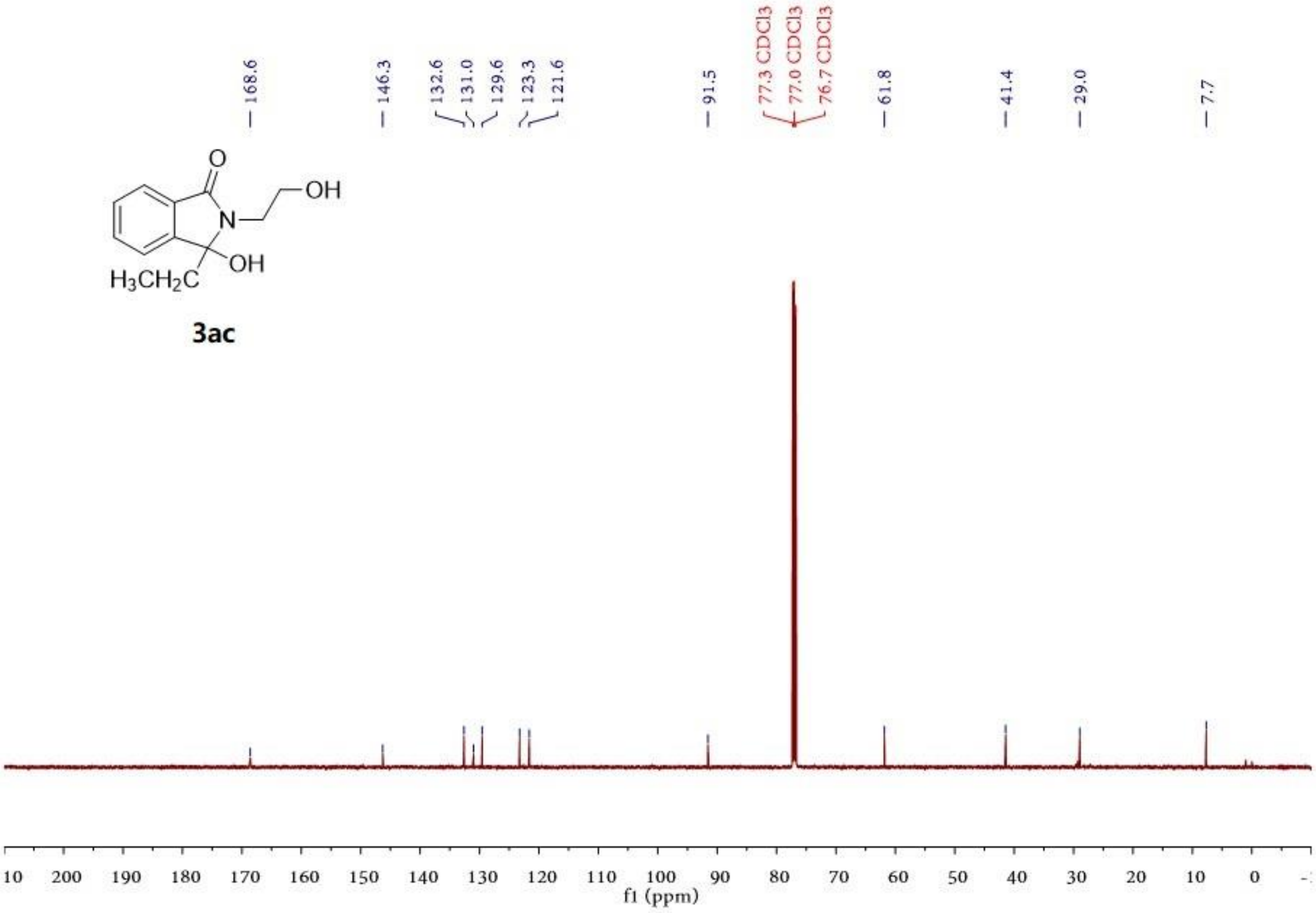
7.56
7.50
7.49
7.47
7.46
7.44
7.26
3.97
3.96
3.95
3.94
3.93
3.92
3.83
3.81
3.79
3.78
3.77
3.76
3.28
3.27
3.26
3.24
3.23
2.21
2.20
2.19
2.17
2.14
2.13
2.11
2.10
2.09
0.49
0.47
0.46
O
OH
N
OH
H_3CH_2C
1.00
1.02
2.06
0.86
1.30
2.26
1.10
2.34
3.30
0.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

3ac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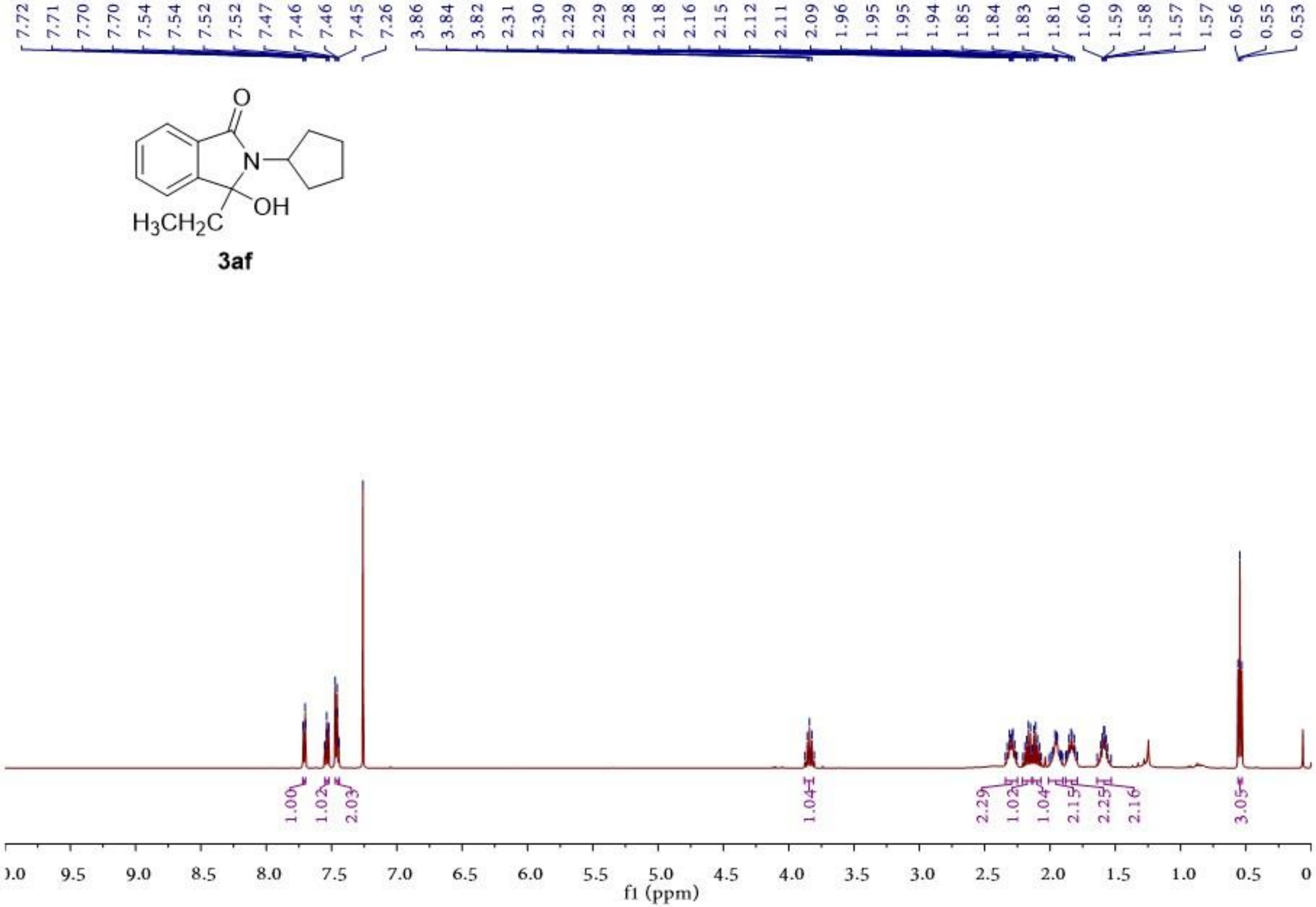
131.0
129.6
123.3
121.6
91.5
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
61.8
41.4
29.0
7.7
O
N
OH
OH
H_3CH_2C
10 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

3ac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7.46
7.45
7.26
3.86
3.84
3.82
2.31
2.30
2.29
2.29
2.28
2.18
2.16
2.15
2.12
2.11
2.09
1.96
1.95
1.95
1.94
1.85
1.84
1.83
1.81
1.60
1.59
1.58
1.57
1.57
0.56
0.55
0.53
O
N
OH
H3CH2C
3af
1.00
1.02
2.03
1.04
2.29
1.02
1.04
2.15
2.25
2.16
3.05
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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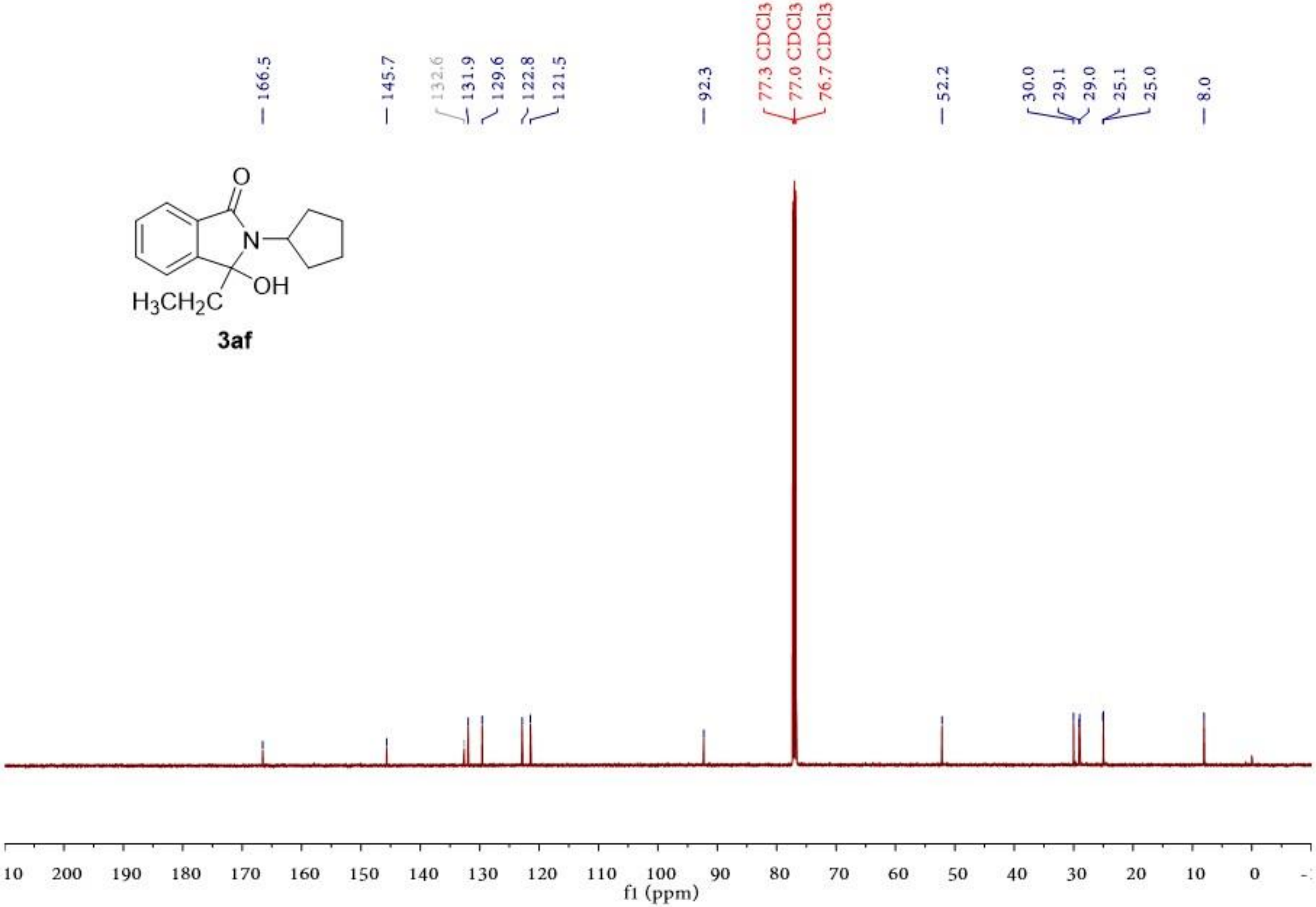
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0
f1 (ppm)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122.8
121.5
92.3
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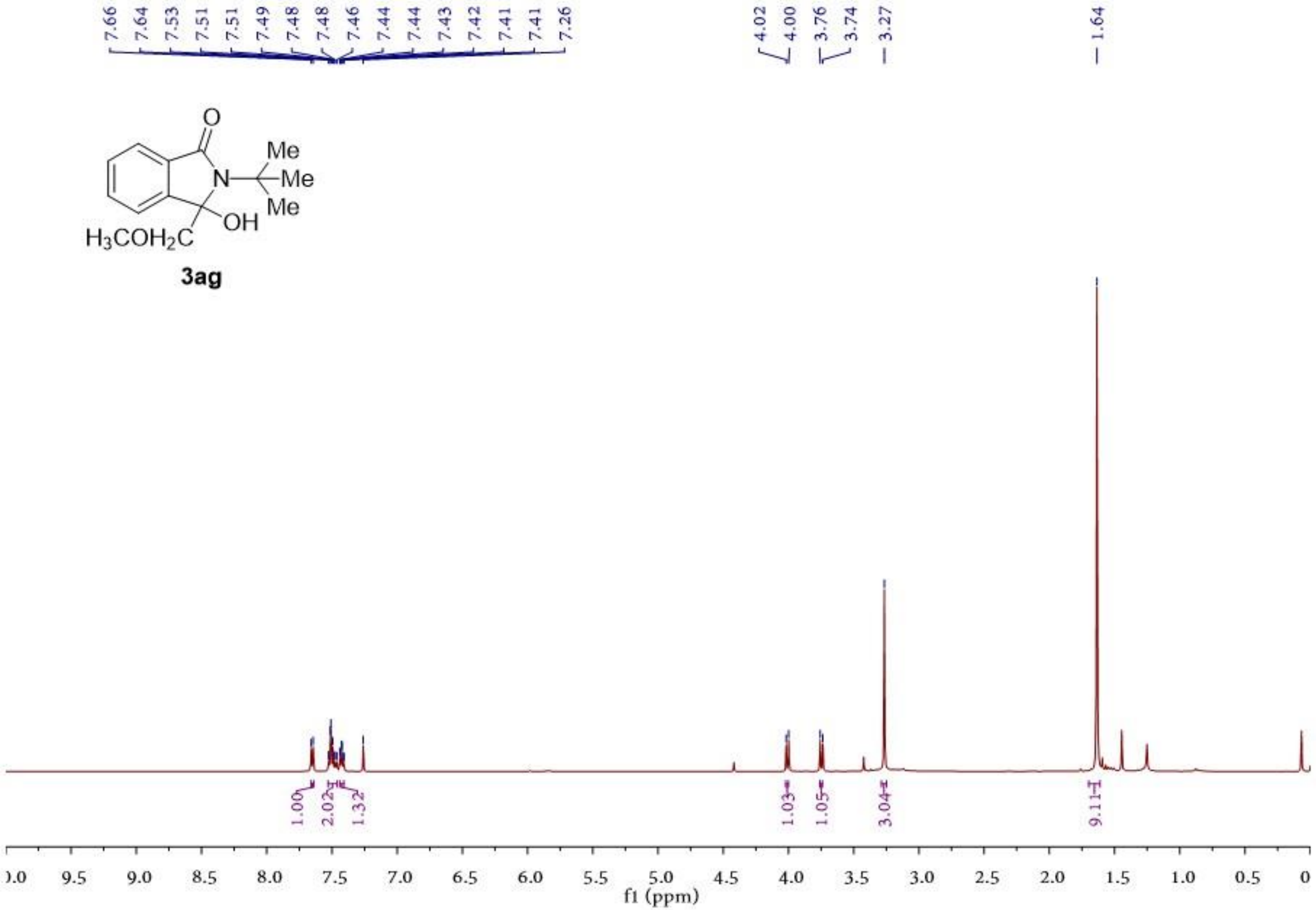
77.0 CDCl3
76.7 CDCl3
52.2
30.0
29.1
29.0
25.1
25.0
8.0
O
N
OH
H3CH2C
3af
10
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.42
7.41
7.41
7.26
4.02
4.00
3.76
3.74
3.27
1.64
O
N
Me
Me
Me
OH
H3COH2C
3ag
1.00
2.02
1.32
1.03
1.05
3.04
9.11
).0
9.5
9.0
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8.0
7.5
7.0
6.5
6.0
5.5
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4.0
3.5
3.0
2.5
2.0
1.5
1.0
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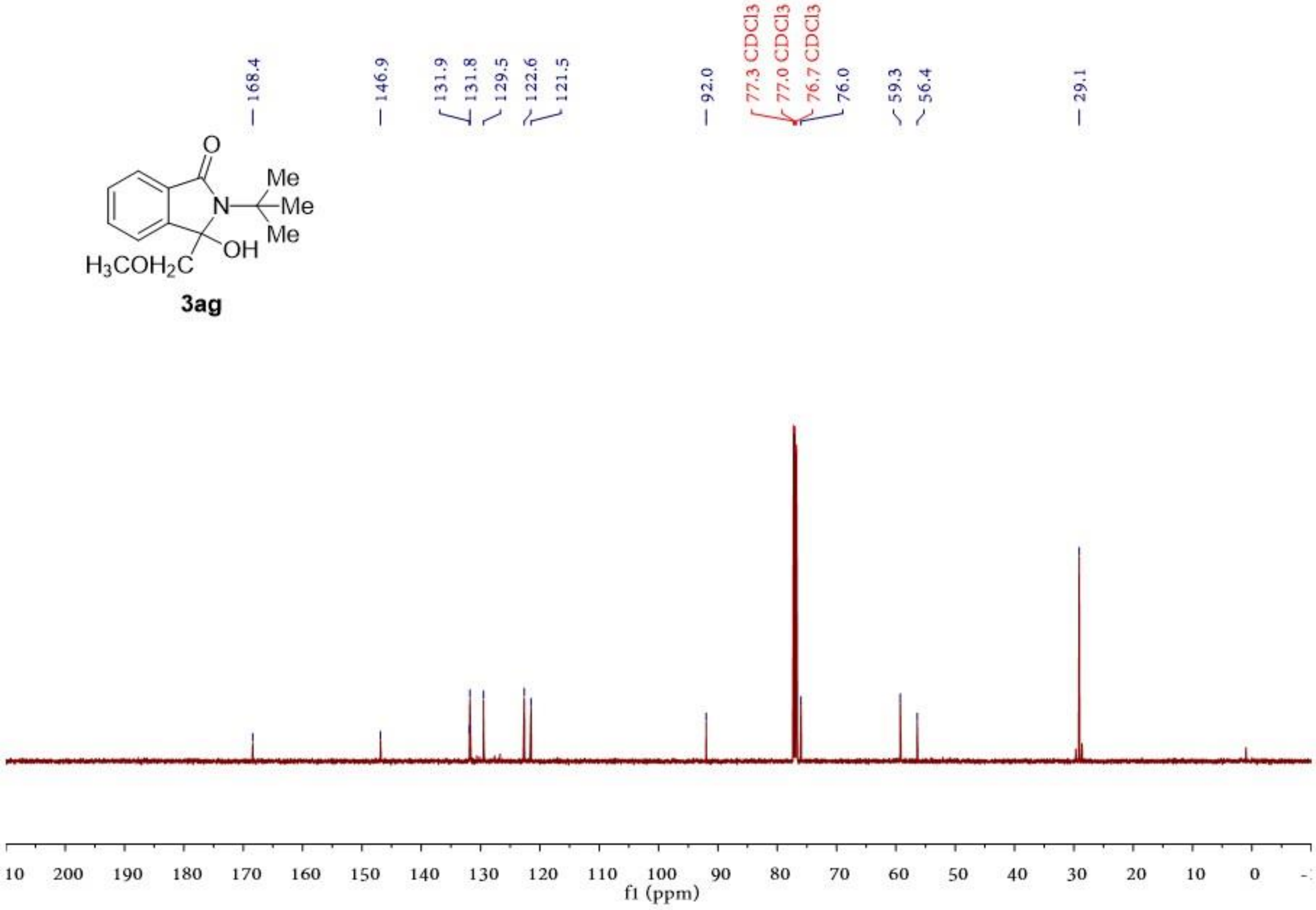
f1 (ppm)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129.5
122.6
121.5
92.0
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77.0 CDCl3
76.7 CDCl3
76.0
59.3
56.4
29.1
O
N
Me
Me
Me
OH
H_3COH_2C
10
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
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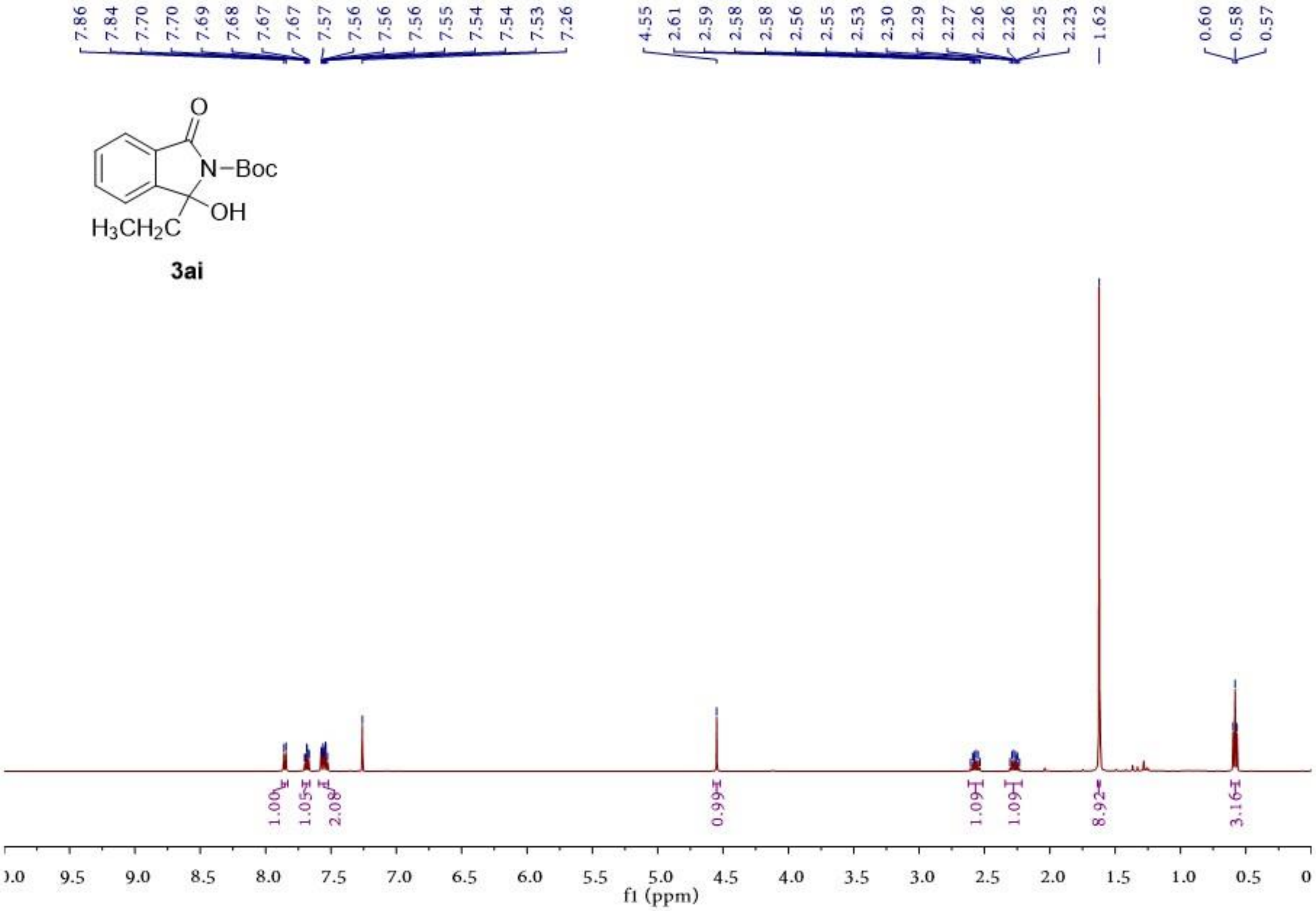
20
10
0
-:
f1 (ppm)

3ag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7.56
7.56
7.55
7.54
7.54
7.53
7.26
4.55
2.61
2.59
2.58
2.58
2.56
2.55
2.53
2.30
2.29
2.27
2.26
2.26
2.25
2.23
1.62
0.60
0.58
0.57
O
N−Boc
OH
H_3CH_2C
3ai
1.00
1.05
2.08
0.99
1.09
1.09
8.92
3.16
0.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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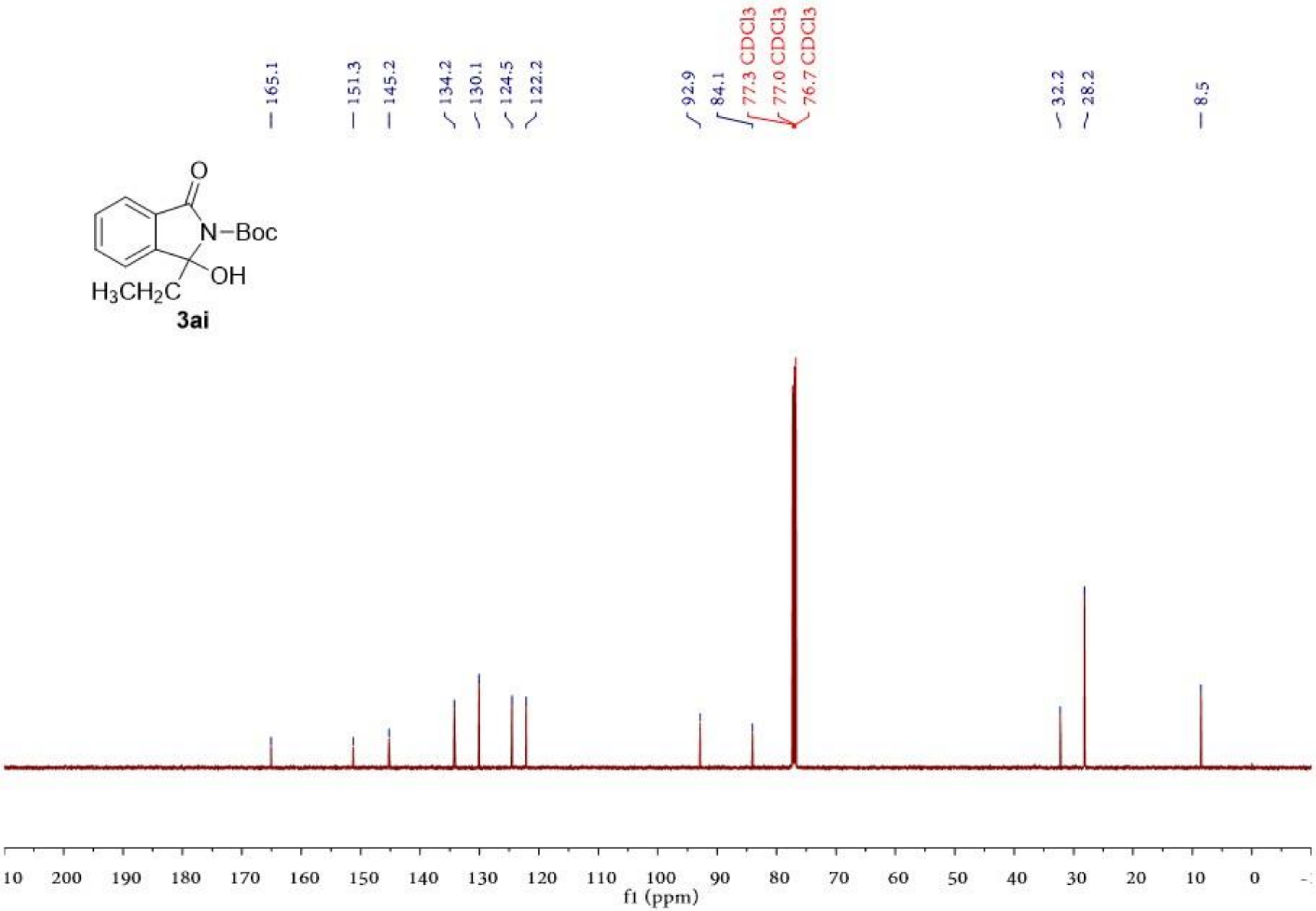
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0
f1 (ppm)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151.3
145.2
134.2
130.1
124.5
122.2
92.9
84.1
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
32.2
28.2
8.5
O
N−Boc
OH
H_3CH_2C
3ai
10
200
190
180
170
160
150
140
130
120
110
100
90
80
70
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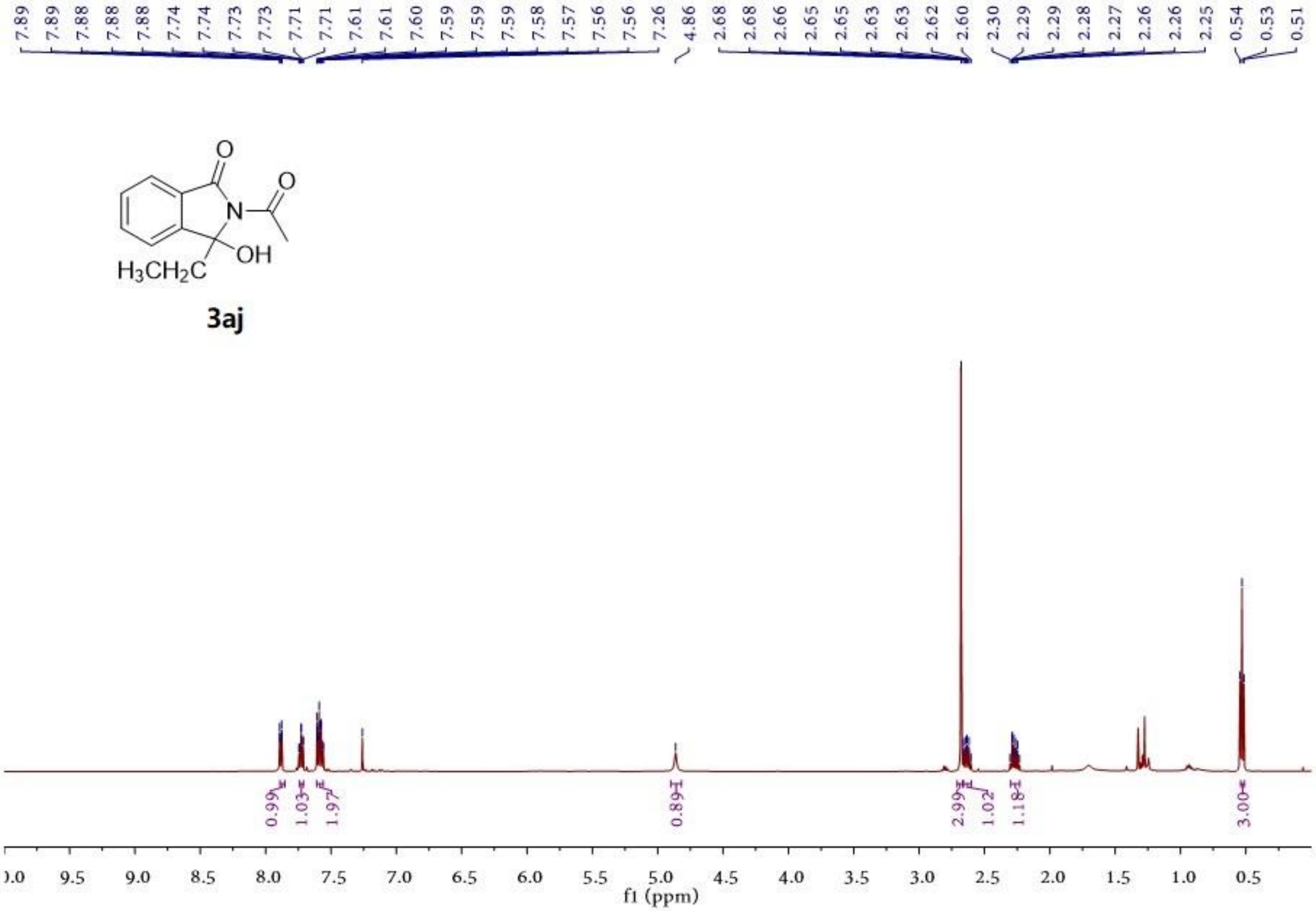
50
40
30
20
10
0
-:
f1 (ppm)



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7.89
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7.88
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7.74
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7.71
7.71
7.61
7.61
7.60
7.59
7.59
7.59
7.58
7.57
7.56
7.56
7.26
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2.68
2.66
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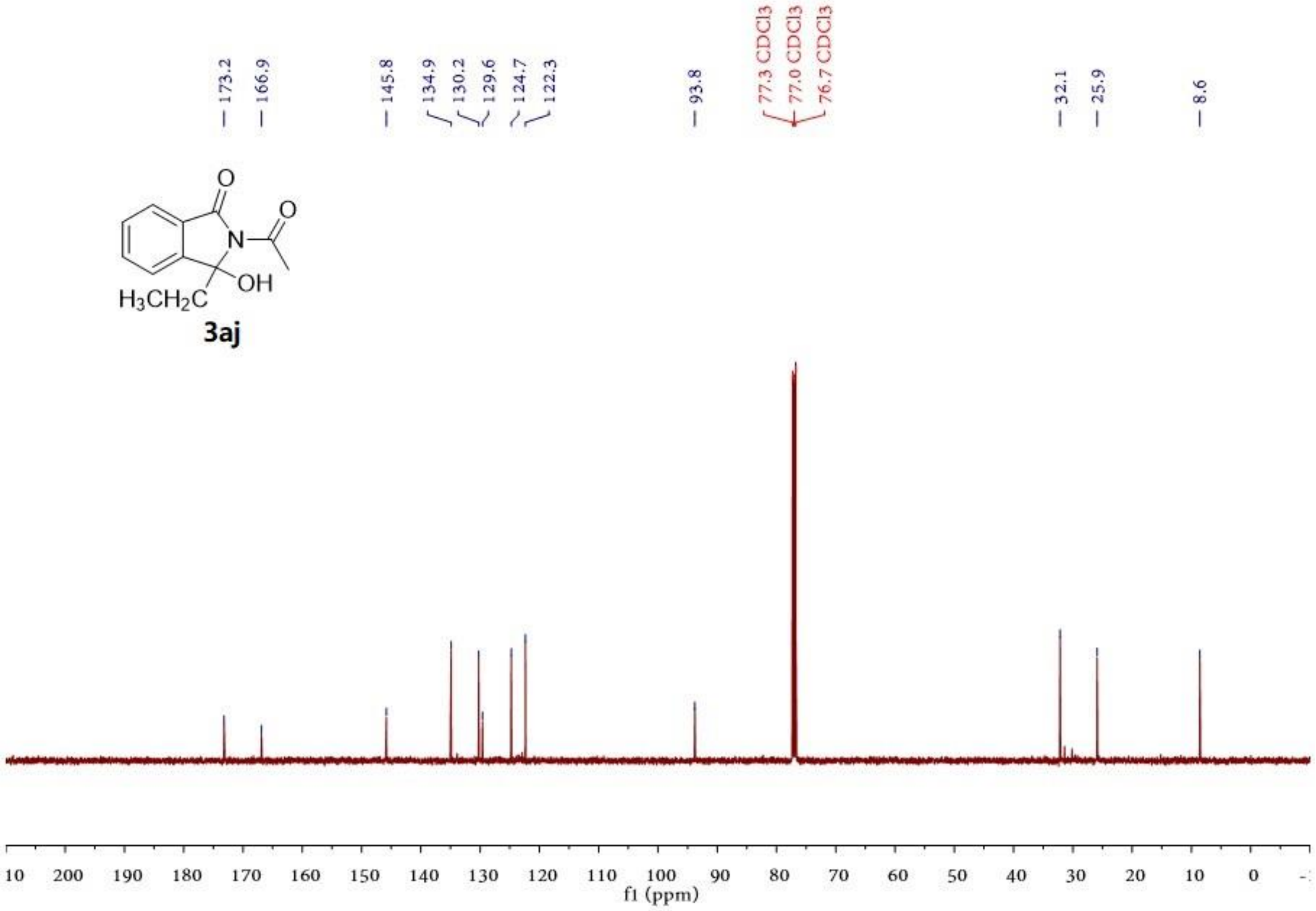
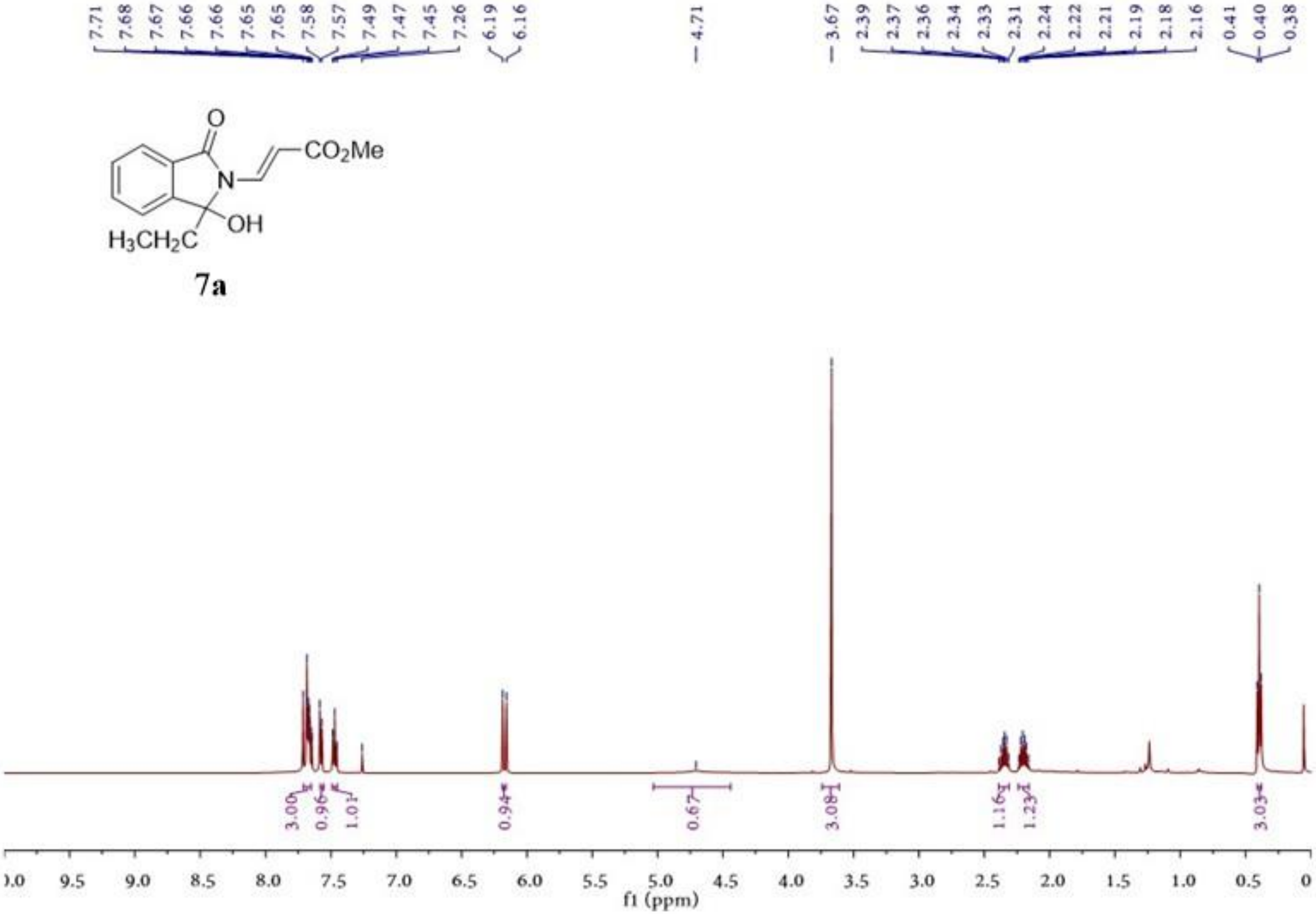
2.63
2.62
2.60
2.30
2.29
2.29
2.28
2.27
2.26
2.26
2.25
0.54
0.53
0.51
O
O
N
OH
H3CH2C
3aj
0.99
1.03
1.97
0.89
2.99
1.02
1.18
3.00
).0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)


173.2
166.9
145.8
134.9
130.2
129.6
124.7
122.3
93.8
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
32.1
25.9
8.6
O
O
N
OH
H_3CH_2C
3aj
10
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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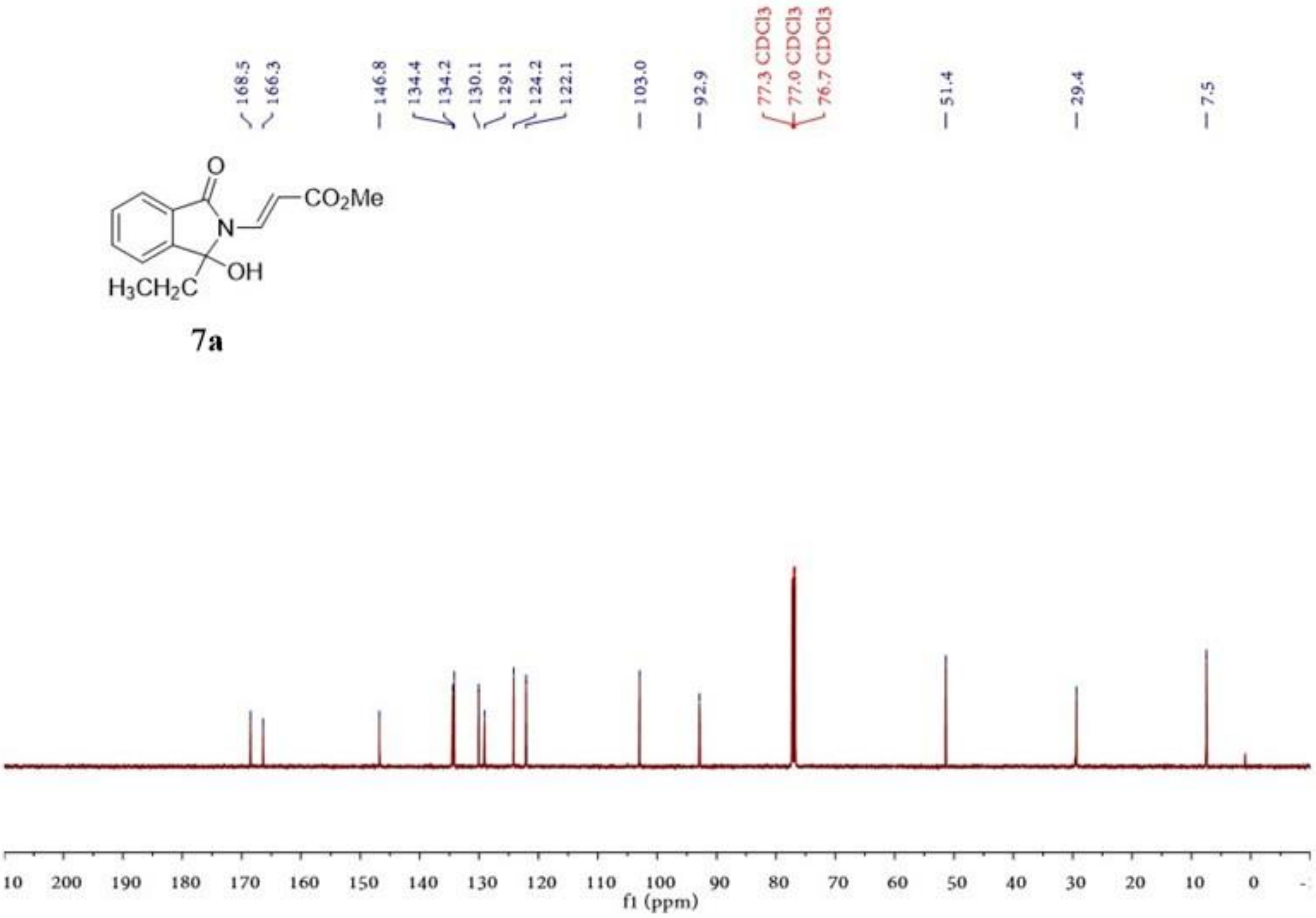
7.68
7.67
7.66
7.66
7.65
7.65
7.58
7.57
7.49
7.47
7.45
7.26
6.19
6.16
4.71
3.67
2.39
2.37
2.36
2.34
2.33
2.31
2.24
2.22
2.21
2.19
2.18
2.16
0.41
0.40
0.38
O
CO2Me
N
OH
H3CH2C
7a
3.00
0.96
1.01
0.94
0.67
3.08
1.16
1.23
3.03
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0
f1 (ppm)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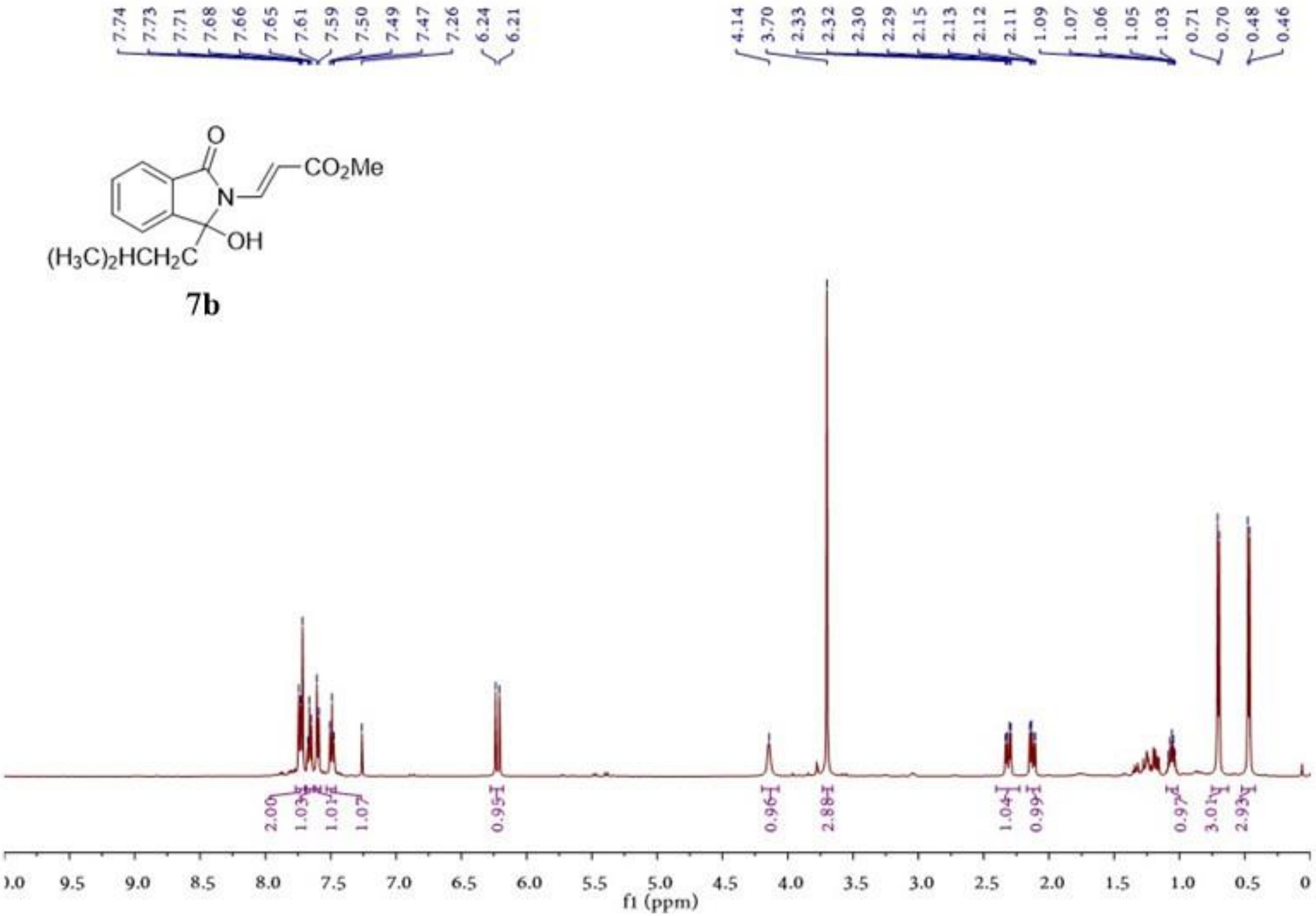
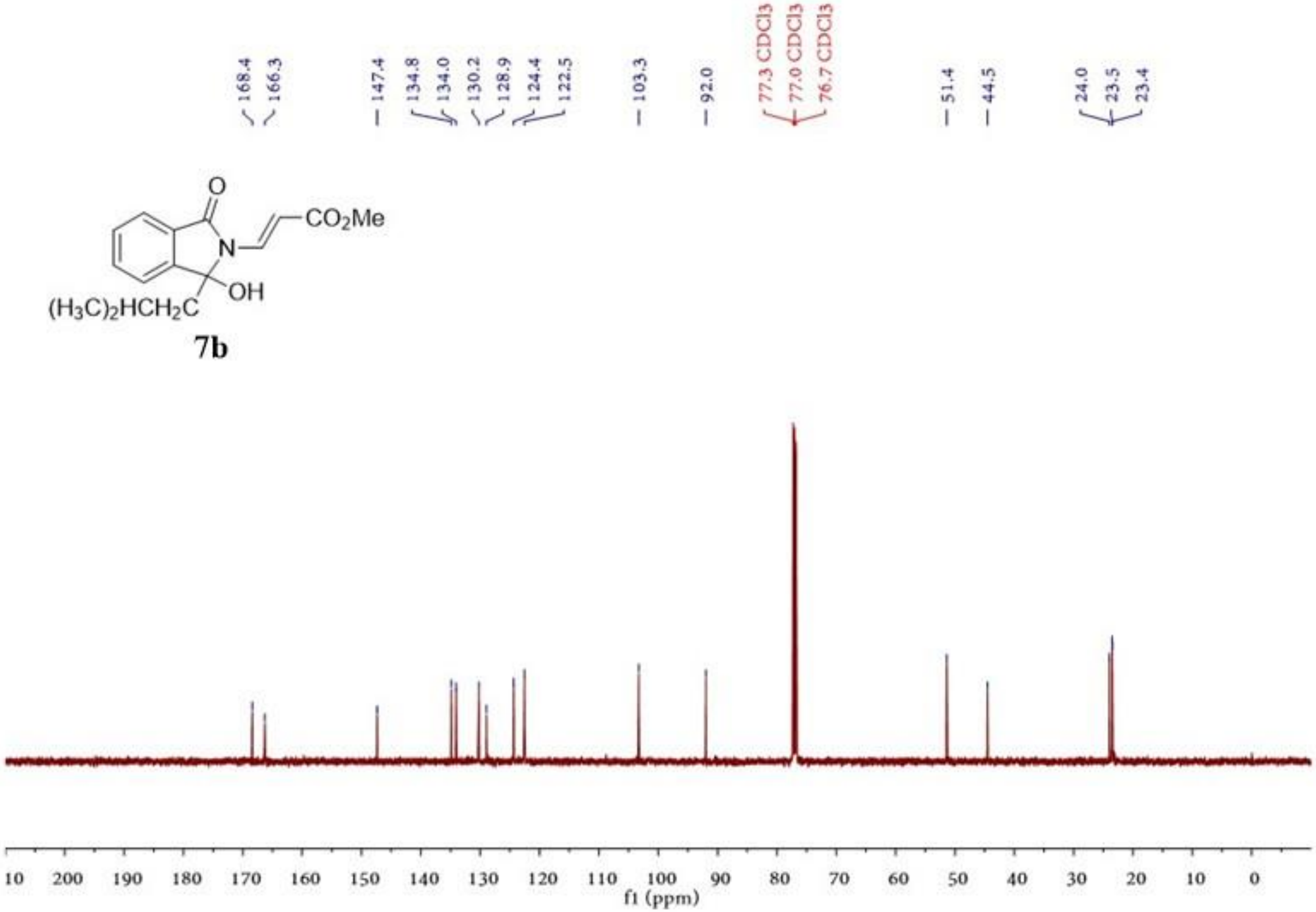
166.3
146.8
134.4
134.2
130.1
129.1
124.2
122.1
103.0
92.9
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
51.4
29.4
7.5
O
N
CO2Me
OH
H3CH2C
7a
10
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-
f1 (ppm)



O
CO2Me
N
OH
(H3C)2HCH2C

7b

7.74
7.73
7.71
7.68
7.66
7.65
7.61
7.59
7.50
7.49
7.47
7.26
6.24
6.21
4.14
3.70
2.33
2.32
2.30
2.29
2.15
2.13
2.12
2.11
1.09
1.07
1.06
1.05
1.03
0.71
0.70
0.48
0.46
2.00
1.03
1.01
1.07
0.95
0.96
2.88
1.04
0.99
0.97
3.01
2.93
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0
f1 (ppm)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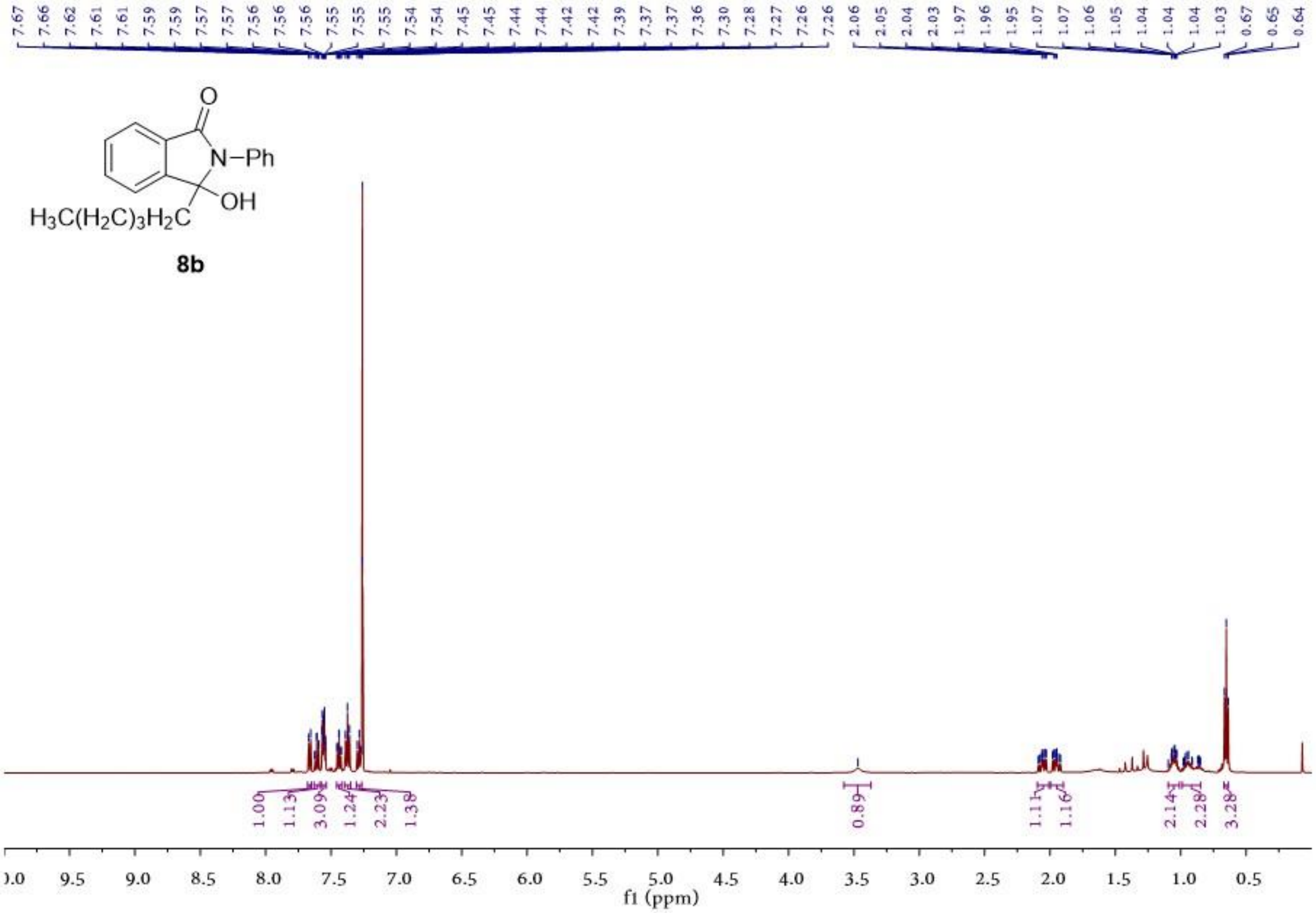
166.3
147.4
134.8
134.0
130.2
128.9
124.4
122.5
103.3
92.0
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
51.4
44.5
24.0
23.5
23.4
O
N
CO2Me
OH
(H3C)2HCH2C
7b
10
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.67
7.66
7.62
7.61
7.61
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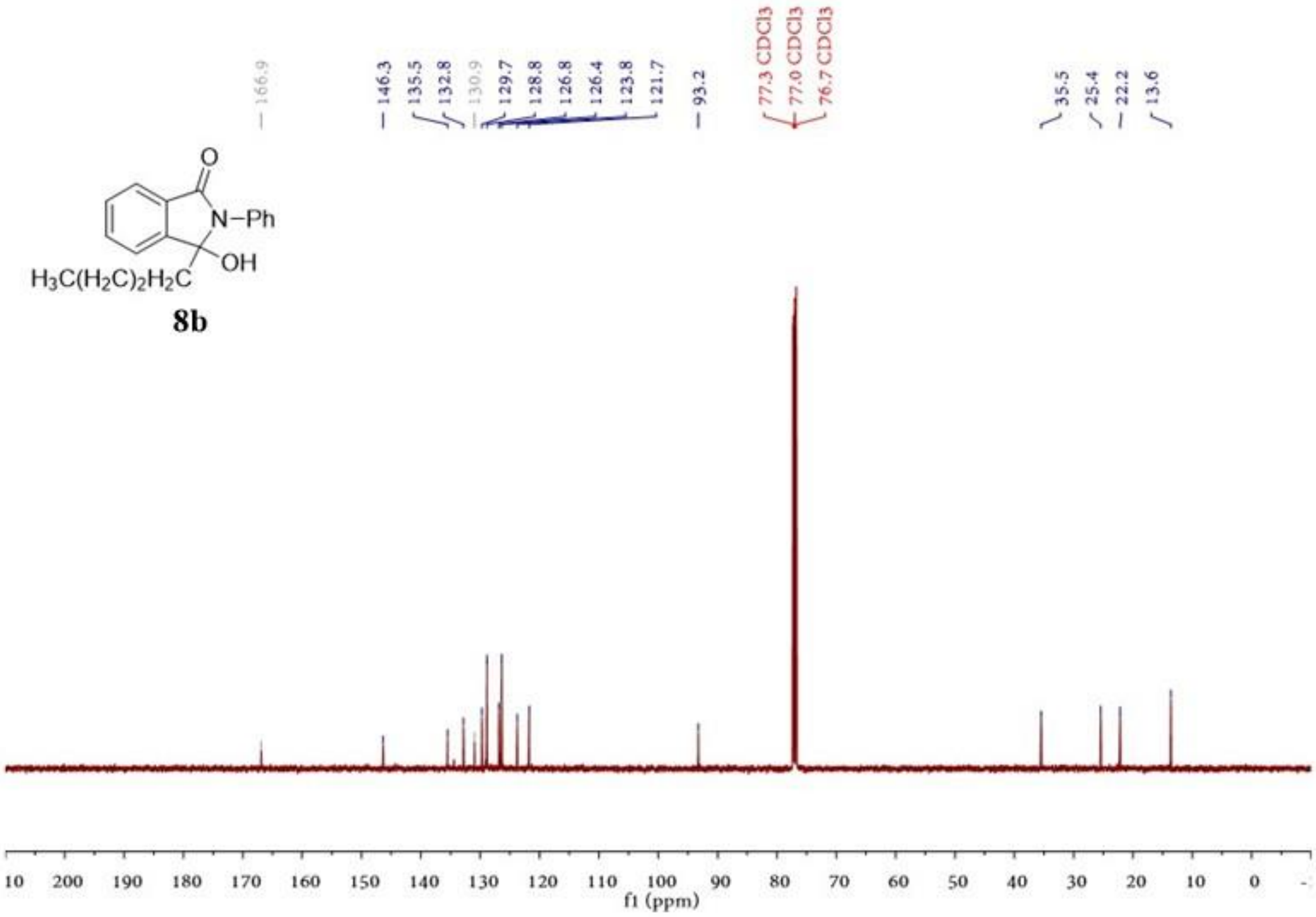
7.59
7.57
7.57
7.56
7.56
7.56
7.55
7.55
7.55
7.54
7.54
7.45
7.45
7.44
7.44
7.42
7.42
7.39
7.37
7.37
7.36
7.30
7.28
7.27
7.26
7.26
2.06
2.05
2.04
2.03
1.97
1.96
1.95
1.07
1.07
1.06
1.05
1.04
1.04
1.04
1.03
0.67
0.65
0.64
O
N−Ph
OH
$H_3C(H_2C)_3H_2C$
8b
1.00
1.13
3.09
1.24
2.23
1.38
0.89
1.11
1.16
2.14
2.28
3.28
).0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)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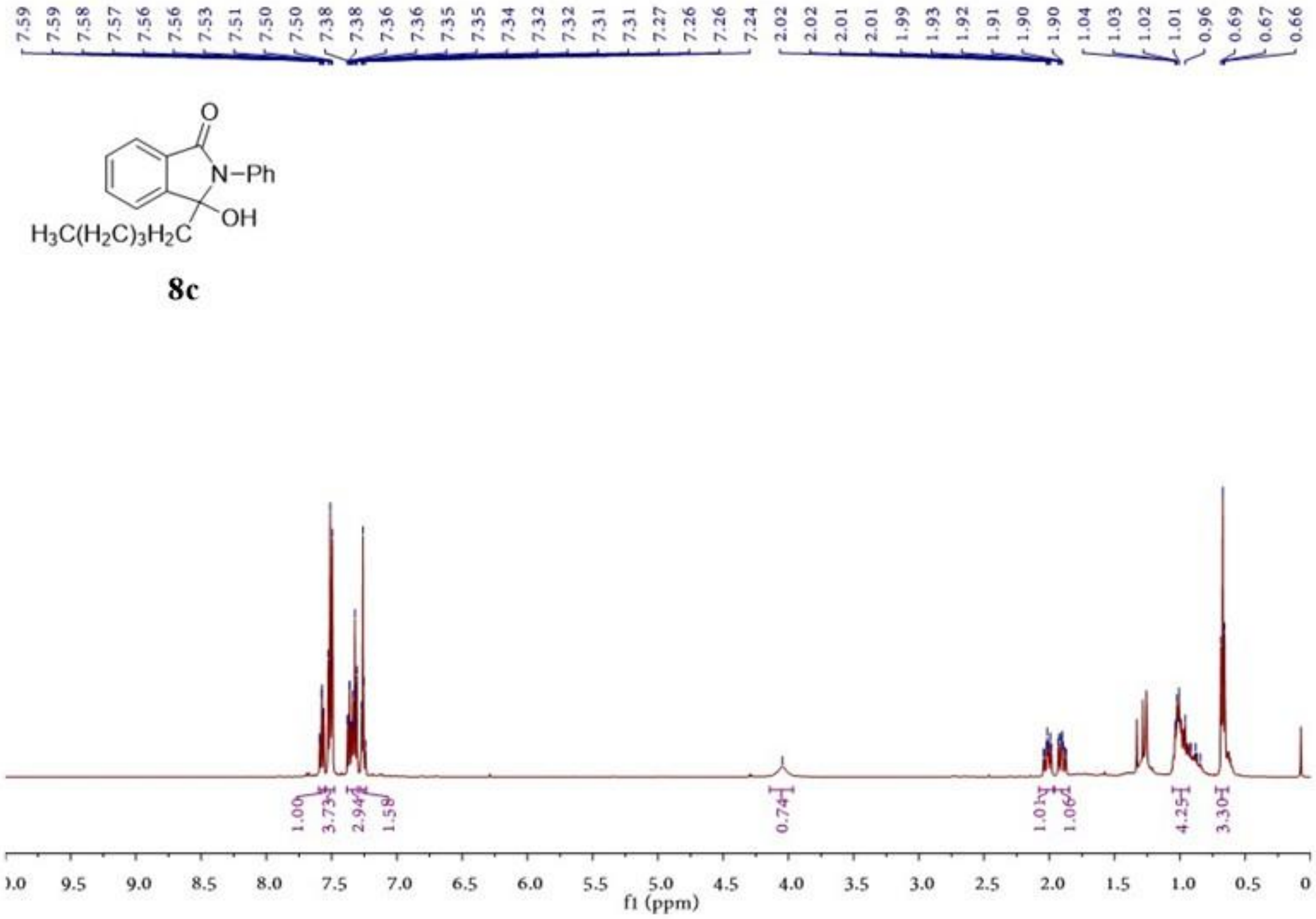
146.3
135.5
132.8
130.9
129.7
128.8
126.8
126.4
123.8
121.7
93.2
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
35.5
25.4
22.2
13.6
O
N−Ph
OH
H3C(H2C)2H2C
10
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-
f1 (ppm)

8b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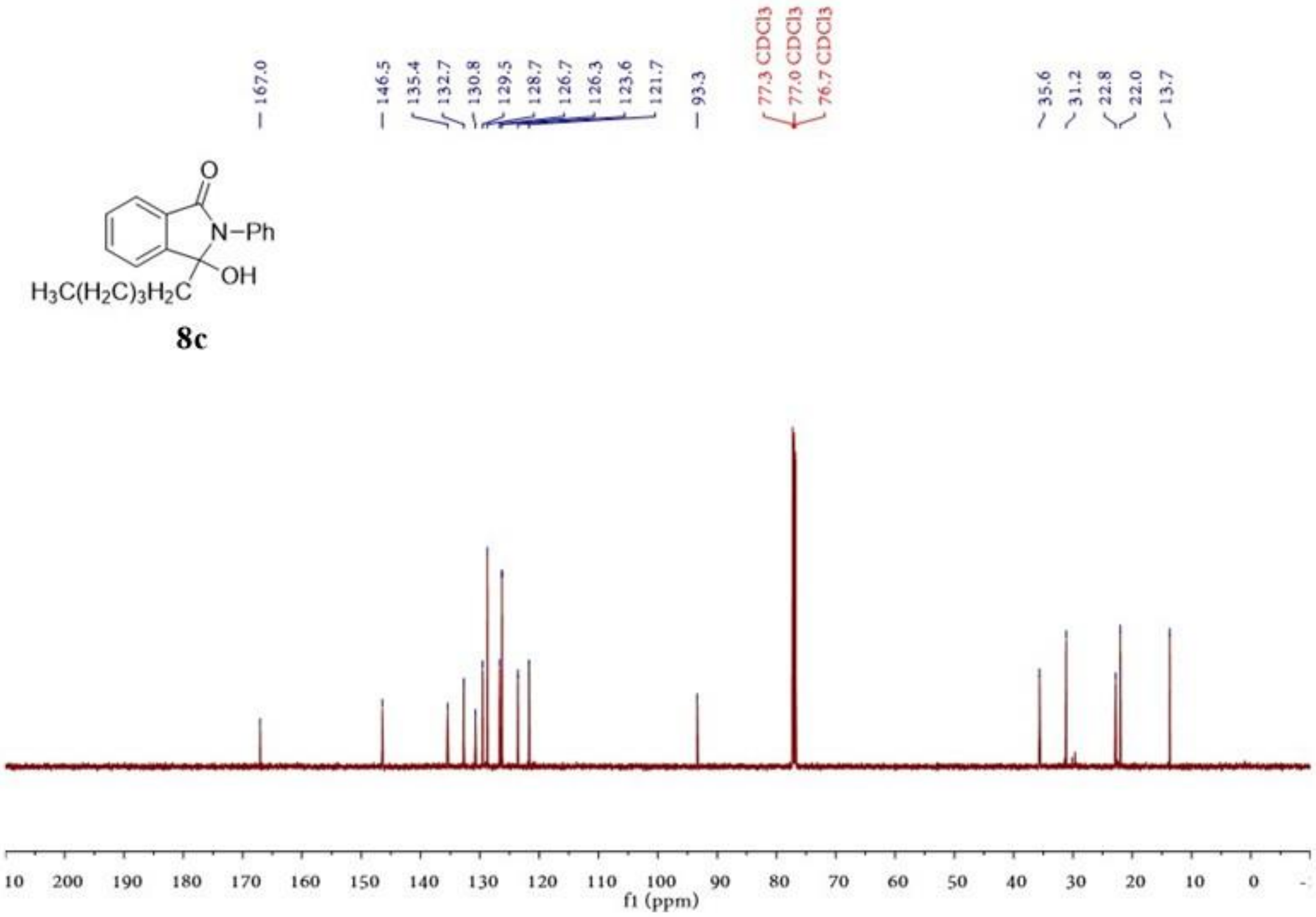
7.59
7.58
7.57
7.56
7.56
7.53
7.51
7.50
7.50
7.38
7.38
7.36
7.36
7.35
7.35
7.34
7.32
7.32
7.31
7.31
7.27
7.26
7.26
7.24
2.02
2.02
2.01
2.01
1.99
1.93
1.92
1.91
1.90
1.90
1.04
1.03
1.02
1.01
0.96
0.69
0.67
0.66
O
N–Ph
OH
H3C(H2C)3H2C
1.00
3.73
2.94
1.58
0.74
1.01
1.06
4.25
3.30
0.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0
f1 (ppm)

8c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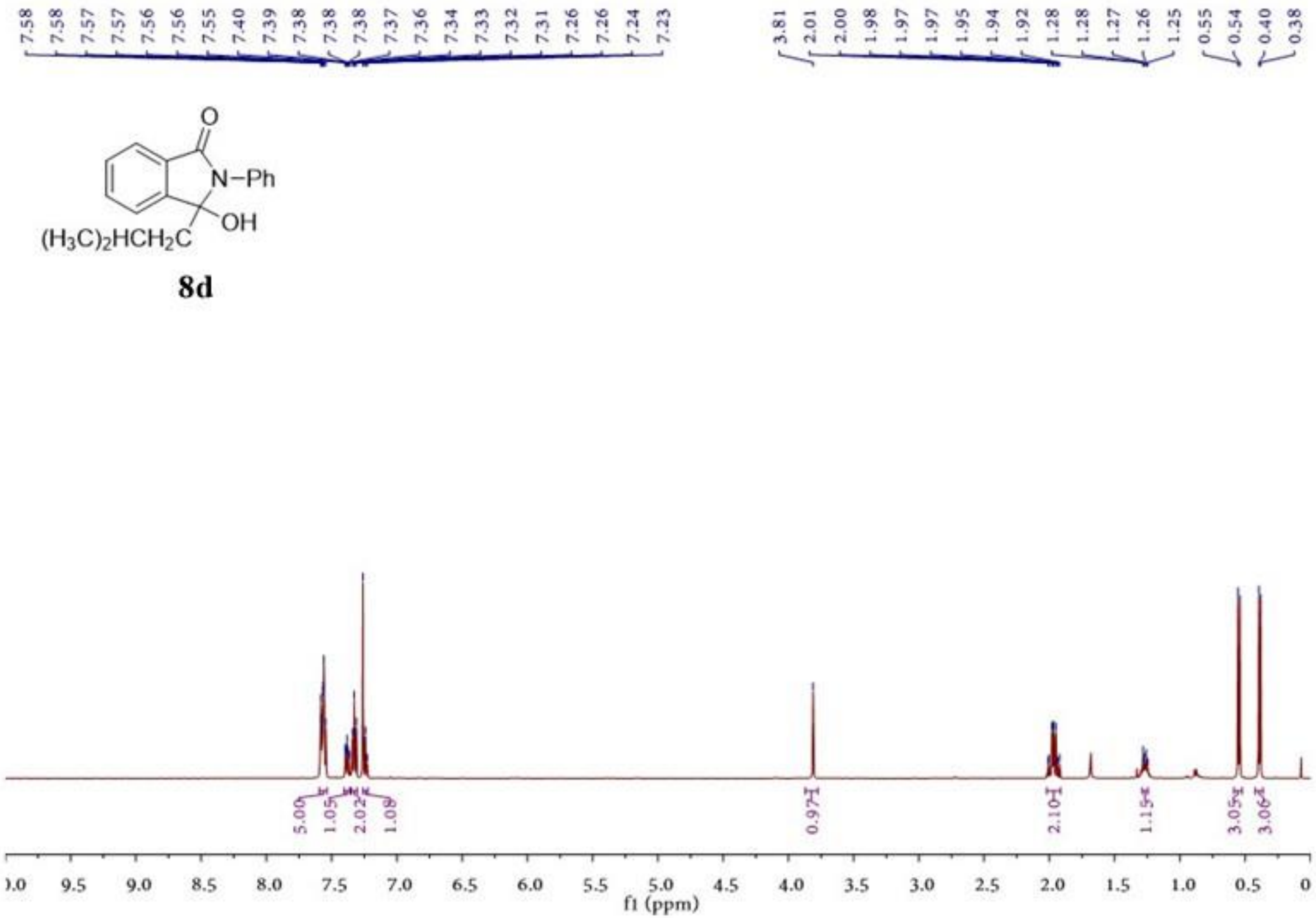
146.5
135.4
132.7
130.8
129.5
128.7
126.7
126.3
123.6
121.7
93.3
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
35.6
31.2
22.8
22.0
13.7
O
N−Ph
OH
H3C(H2C)3H2C
10
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

8c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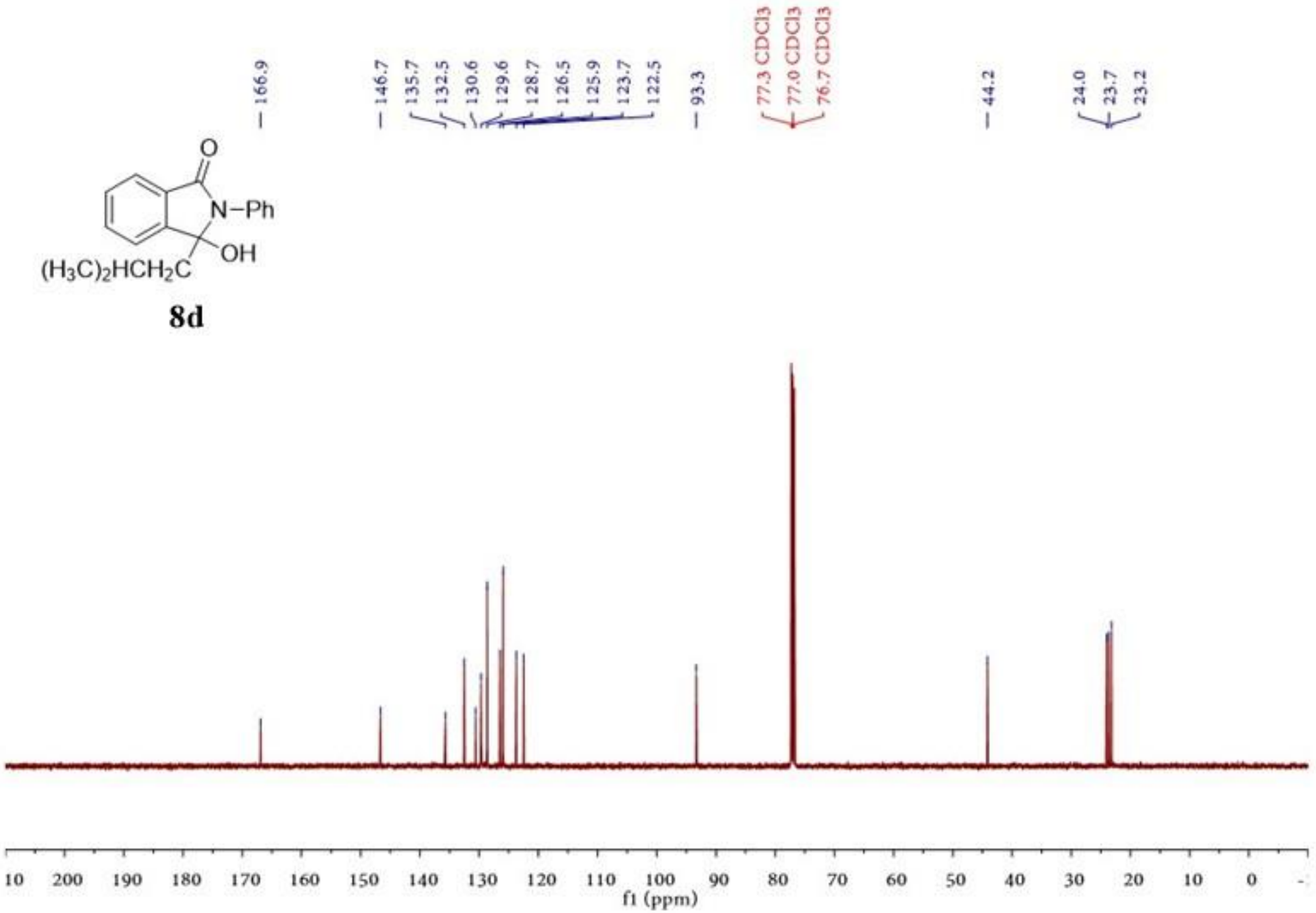
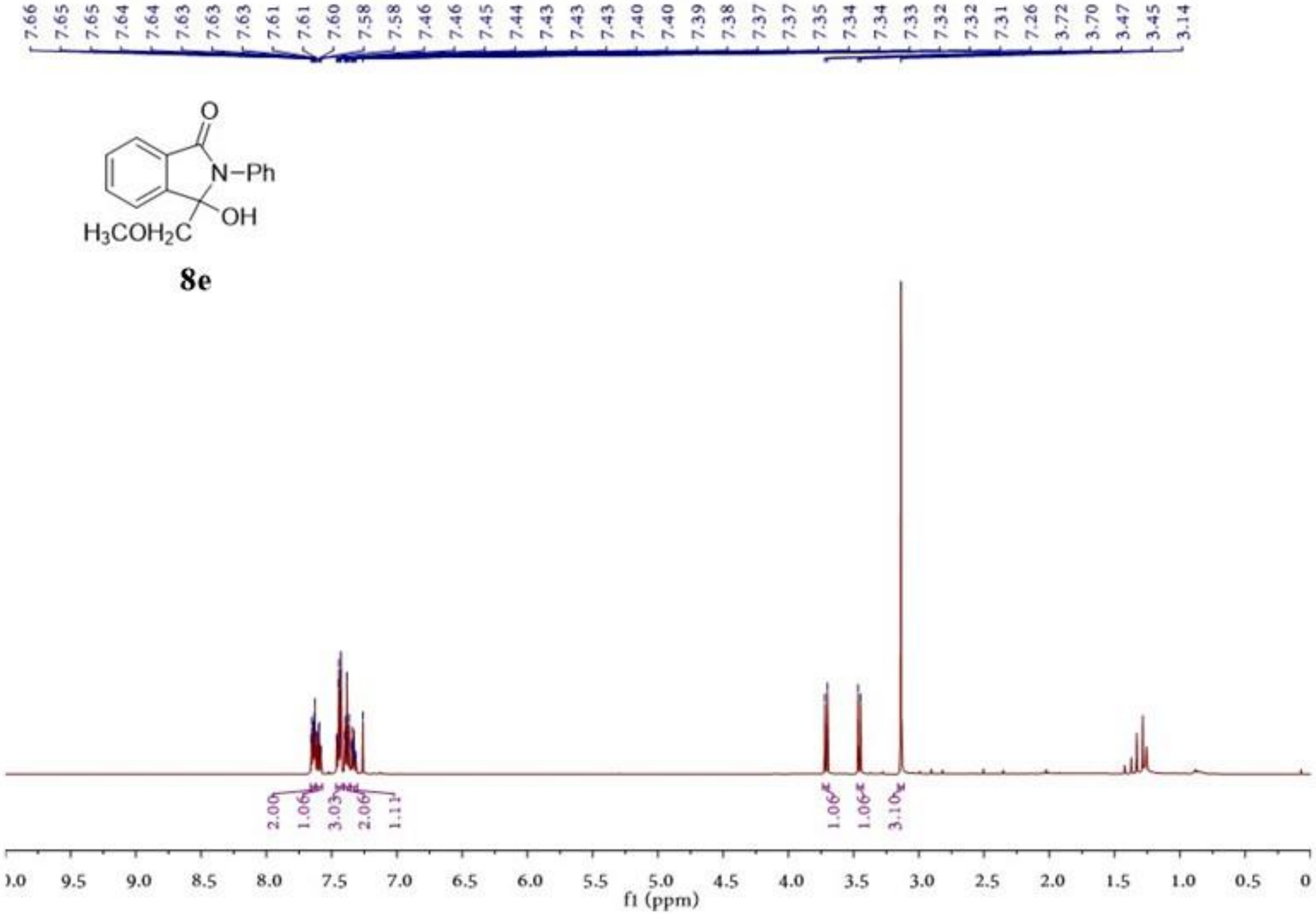
7.58
7.57
7.57
7.56
7.56
7.55
7.40
7.39
7.38
7.38
7.38
7.37
7.36
7.34
7.33
7.32
7.31
7.26
7.26
7.24
7.23
3.81
2.01
2.00
1.98
1.97
1.97
1.95
1.94
1.92
1.28
1.28
1.27
1.26
1.25
0.55
0.54
0.40
0.38
O
N−Ph
OH
(H3C)2HCH2C
5.00
1.05
2.02
1.08
0.97
2.10
1.15
3.05
3.06
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0
f1 (ppm)

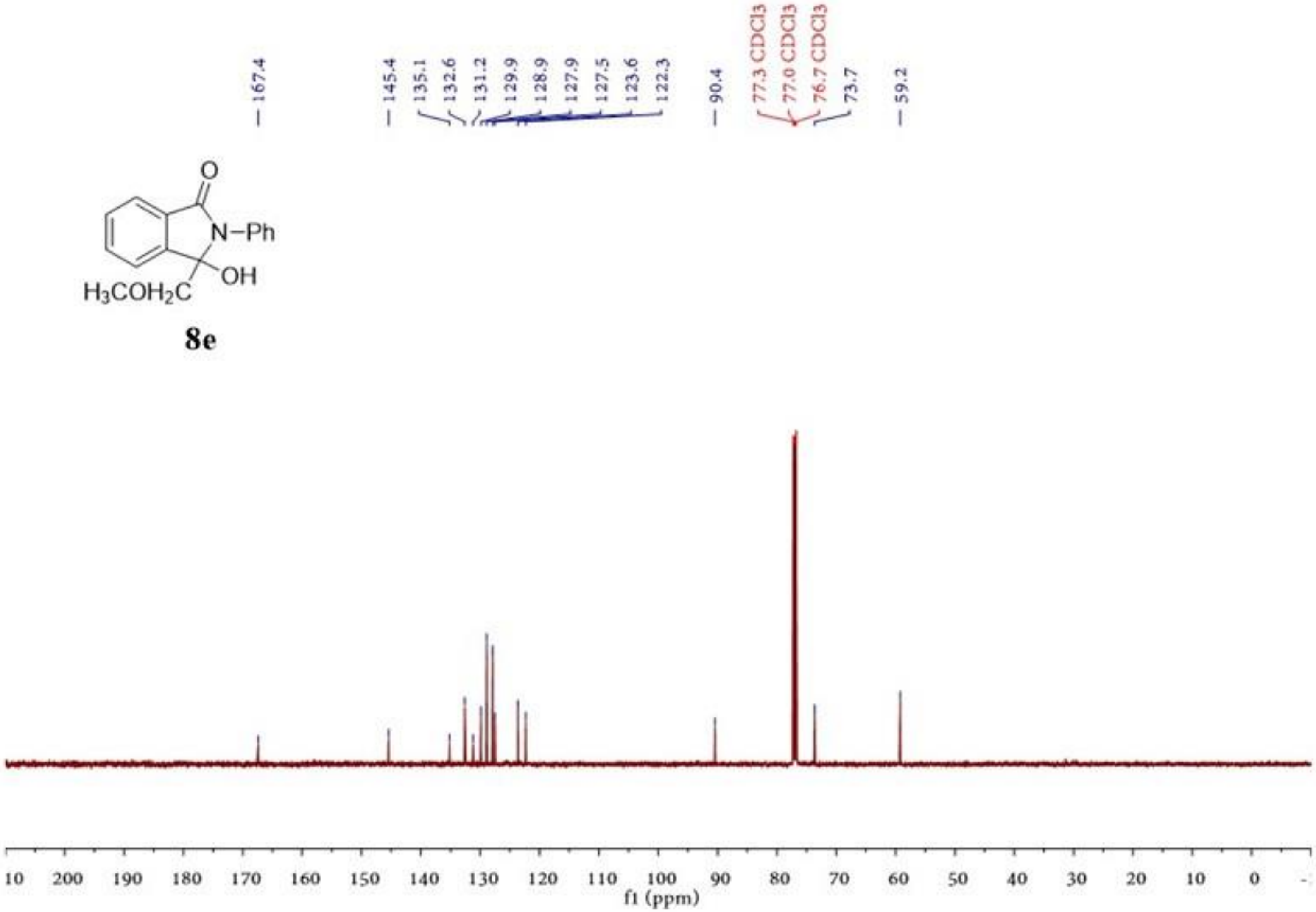
8d

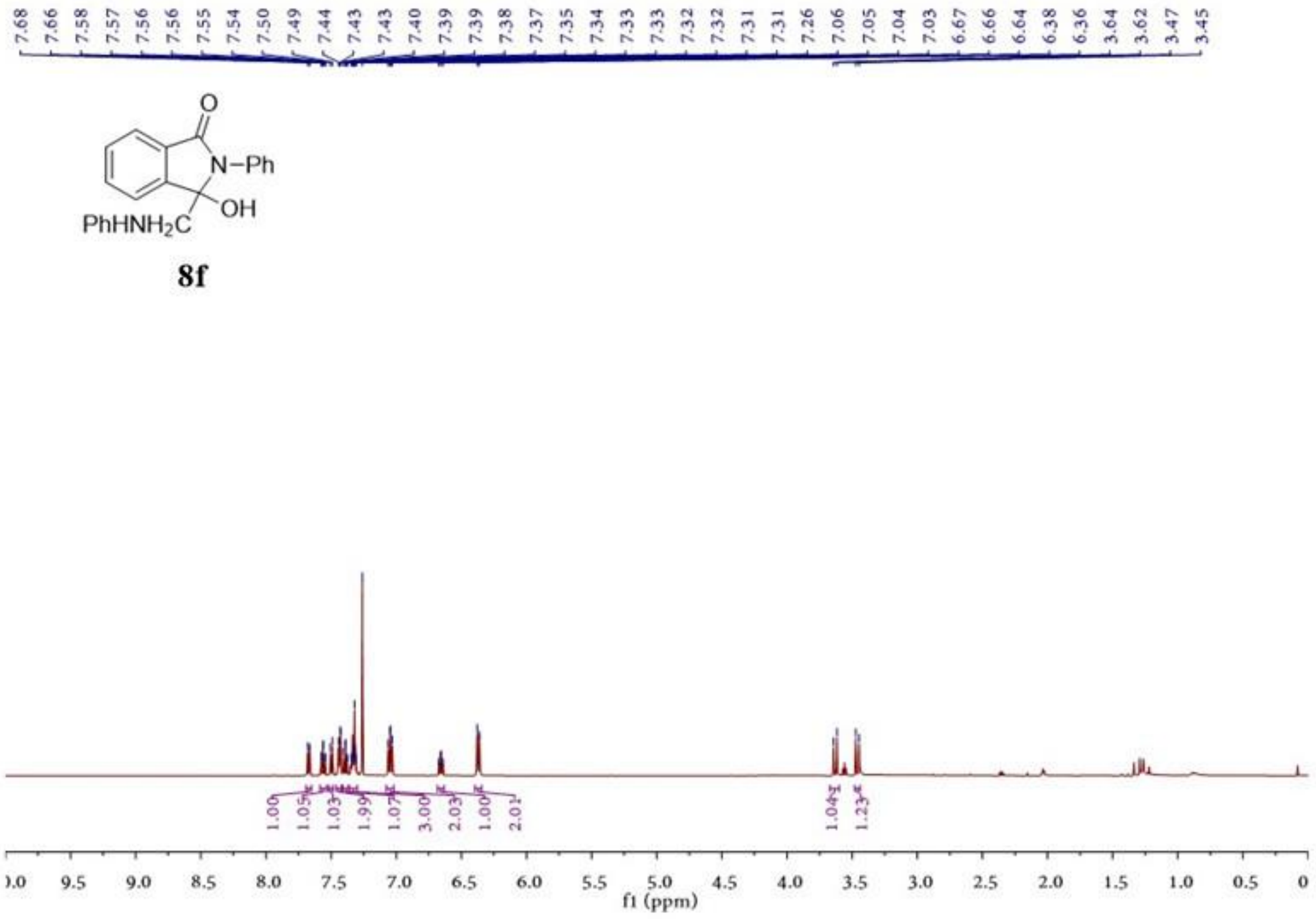

O
N−Ph
OH
(H3C)2HCH2C
166.9
146.7
135.7
132.5
130.6
129.6
128.7
126.5
125.9
123.7
122.5
93.3
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
44.2
24.0
23.7
23.2
10
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

8d


7.66
7.65
7.65
7.64
7.64
7.63
7.63
7.63
7.61
7.61
7.60
7.58
7.58
7.46
7.46
7.45
7.44
7.43
7.43
7.43
7.40
7.40
7.39
7.38
7.37
7.37
7.35
7.34
7.34
7.33
7.32
7.32
7.31
7.26
3.72
3.70
3.47
3.45
3.14
O
N–Ph
OH
H_3COH_2C
2.00
1.06
3.03
2.06
1.11
1.06
1.06
3.10
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0
f1 (ppm)

8e


167.4
145.4
135.1
132.6
131.2
129.9
128.9
127.9
127.5
123.6
122.3
90.4
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
73.7
59.2
O
N–Ph
OH
H3COH2C
8e
10
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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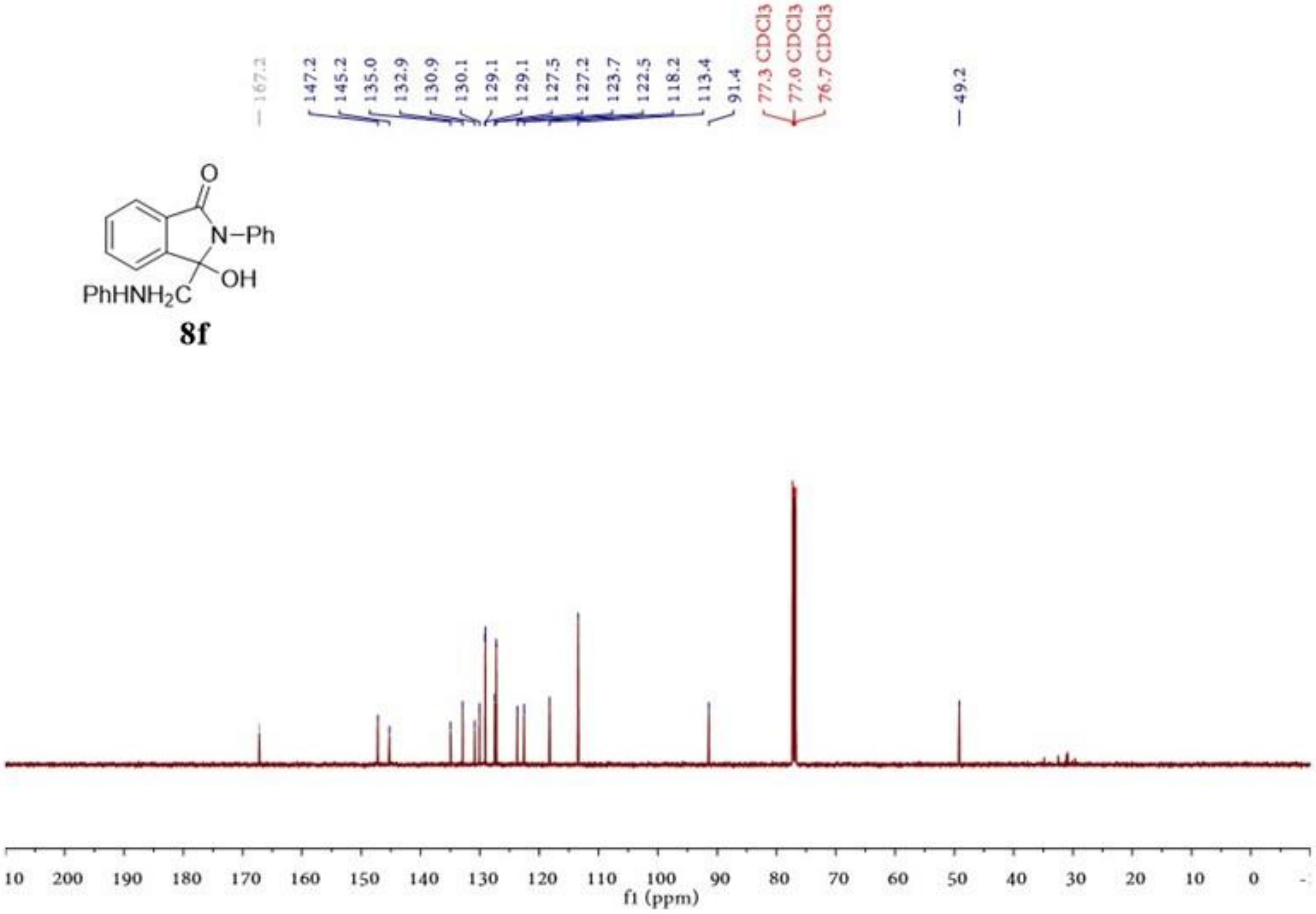
7.49
7.44
7.43
7.43
7.40
7.39
7.39
7.38
7.37
7.35
7.34
7.33
7.33
7.32
7.32
7.31
7.31
7.26
7.06
7.05
7.04
7.03
6.67
6.66
6.64
6.38
6.36
3.64
3.62
3.47
3.45
O
N–Ph
OH
PhHNH$_2$C
1.00
1.05
1.03
1.99
1.07
3.00
2.03
1.00
2.01
1.04
1.23
).0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0
f1 (ppm)

8f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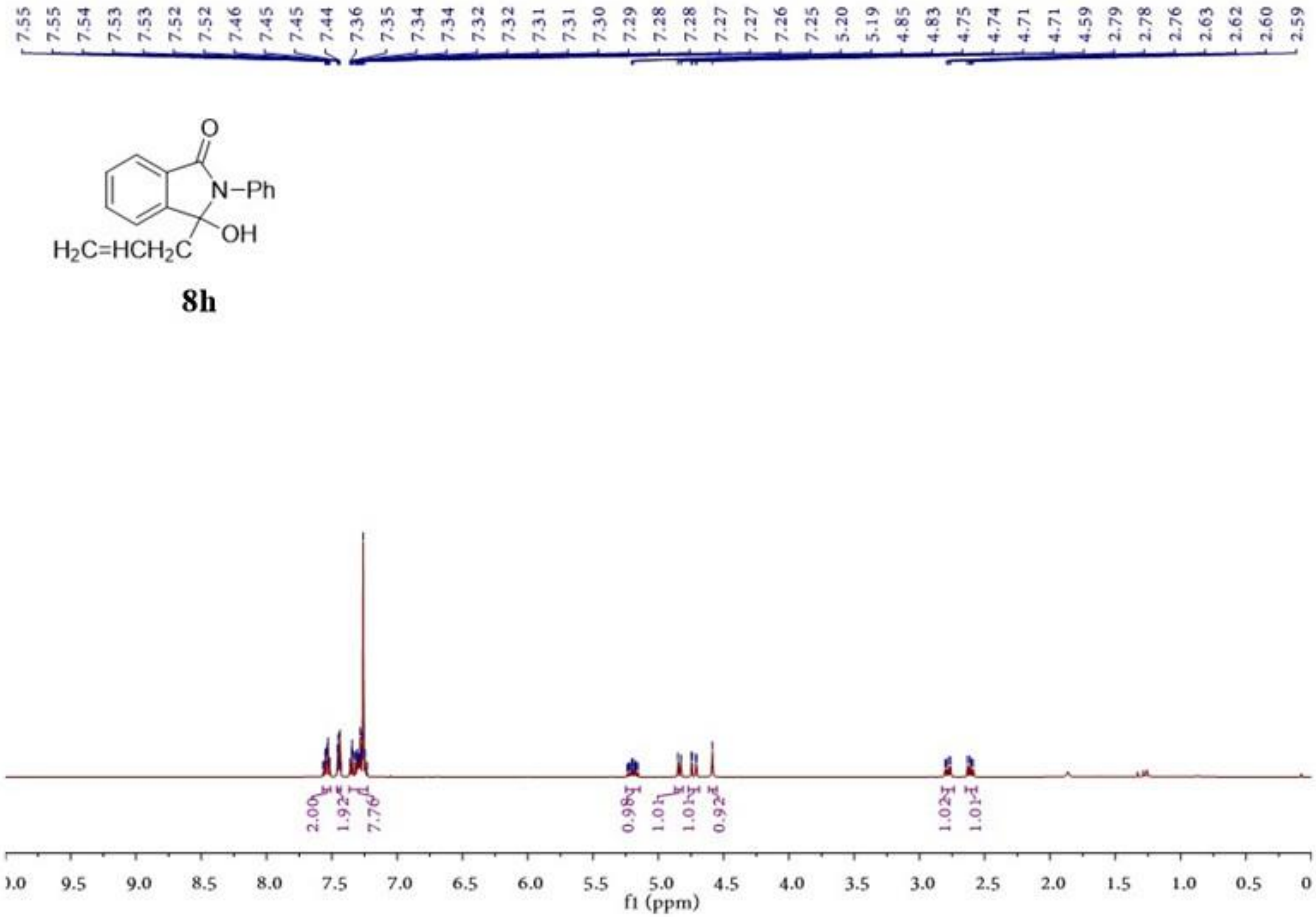
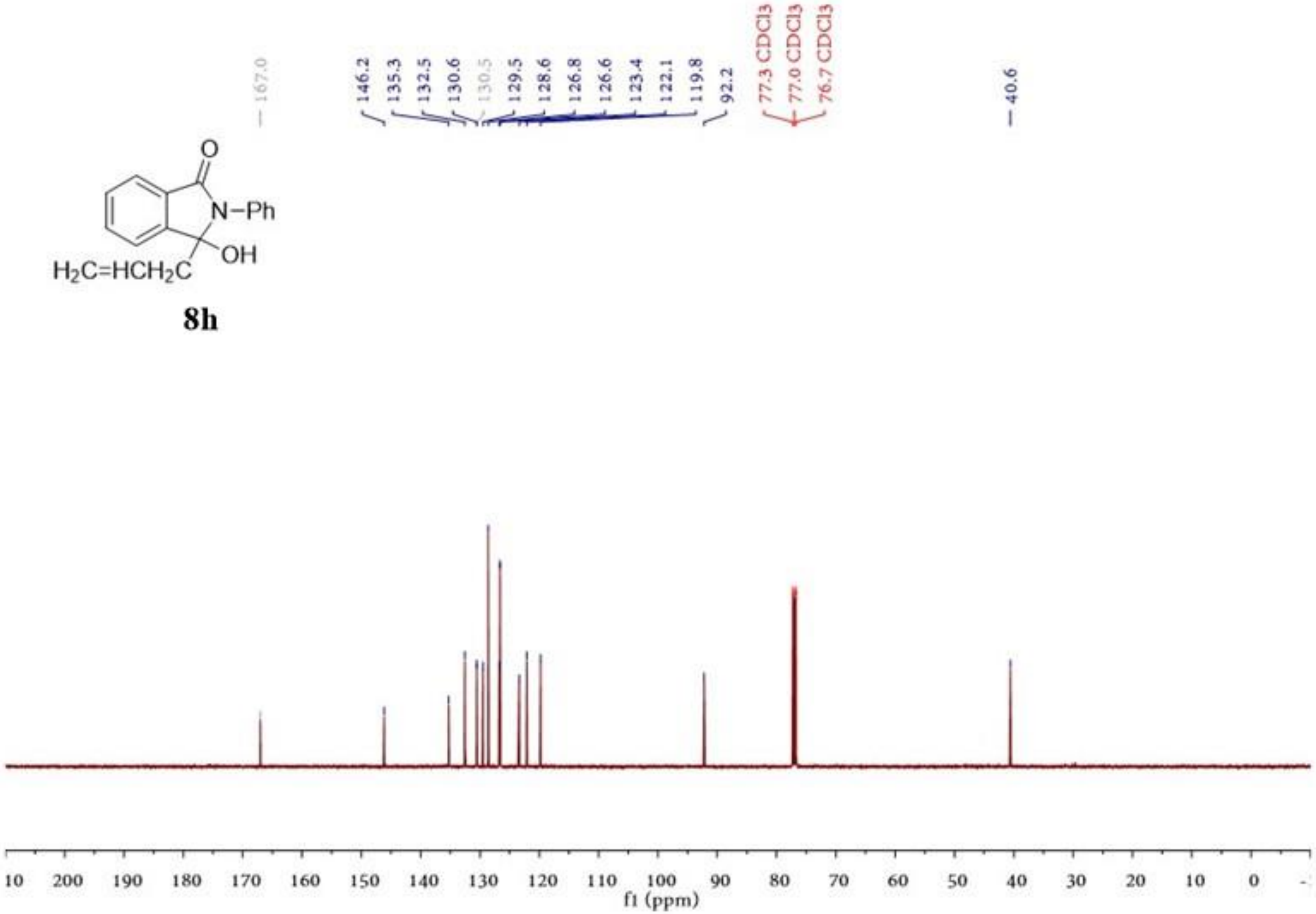
145.2
135.0
132.9
130.9
130.1
129.1
129.1
127.5
127.2
123.7
122.5
118.2
113.4
91.4
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
49.2
O
N−Ph
OH
PhHNH2C
10
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-
f1 (ppm)

8f

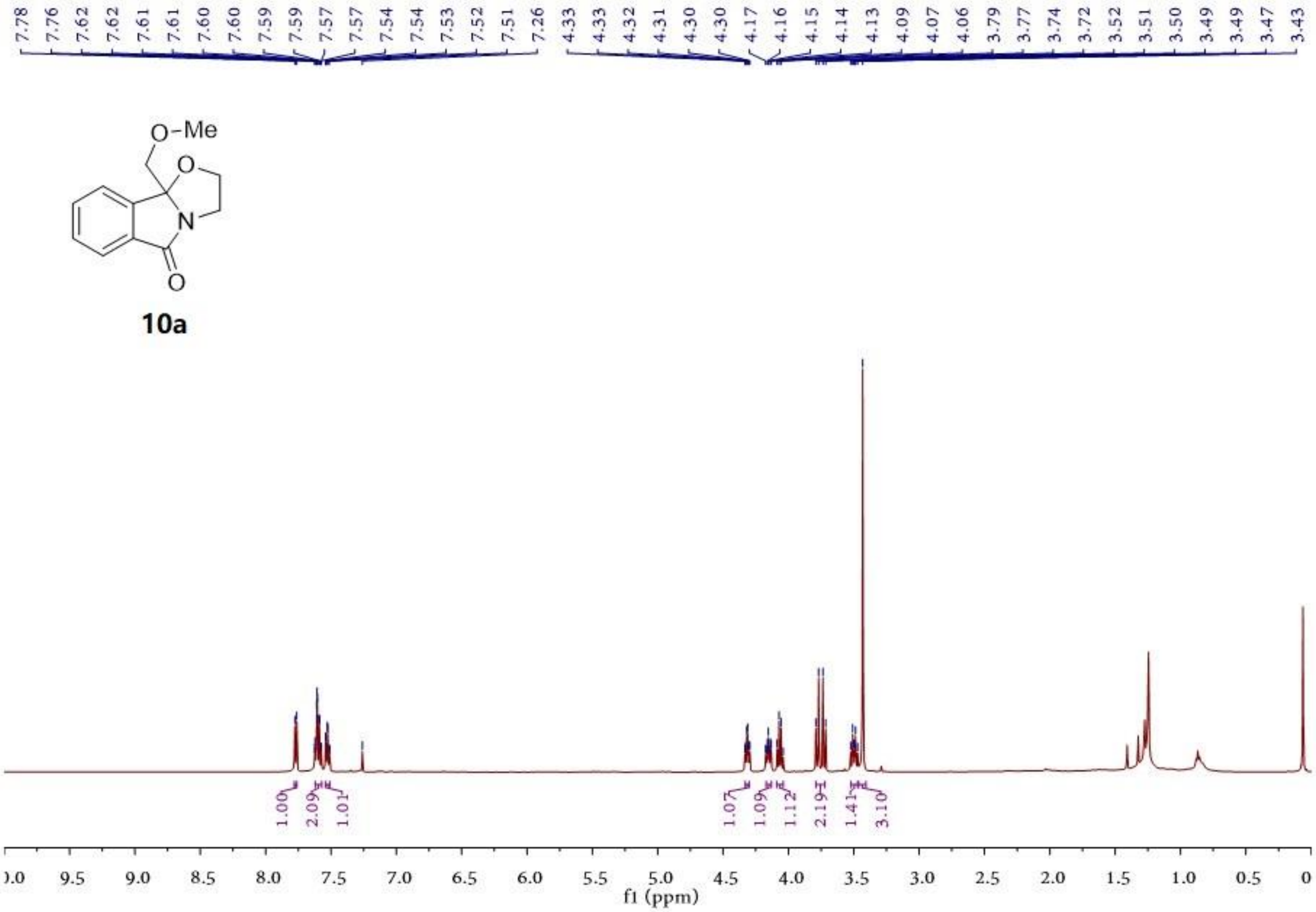

7.55
7.55
7.54
7.53
7.53
7.52
7.52
7.46
7.45
7.45
7.44
7.36
7.35
7.34
7.34
7.32
7.32
7.31
7.31
7.30
7.29
7.28
7.28
7.27
7.27
7.26
7.25
5.20
5.19
4.85
4.83
4.75
4.74
4.71
4.71
4.59
2.79
2.78
2.76
2.63
2.62
2.60
2.59
O
N−Ph
OH
H_2C=HCH_2C
2.00
1.92
7.76
0.98
1.01
1.01
0.92
1.02
1.01
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0
f1 (ppm)

8h


O
N−Ph
OH
$H_2C{=}HCH_2C$

8h

167.0
146.2
135.3
132.5
130.6
130.5
129.5
128.6
126.8
126.6
123.4
122.1
119.8
92.2
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
40.6
10
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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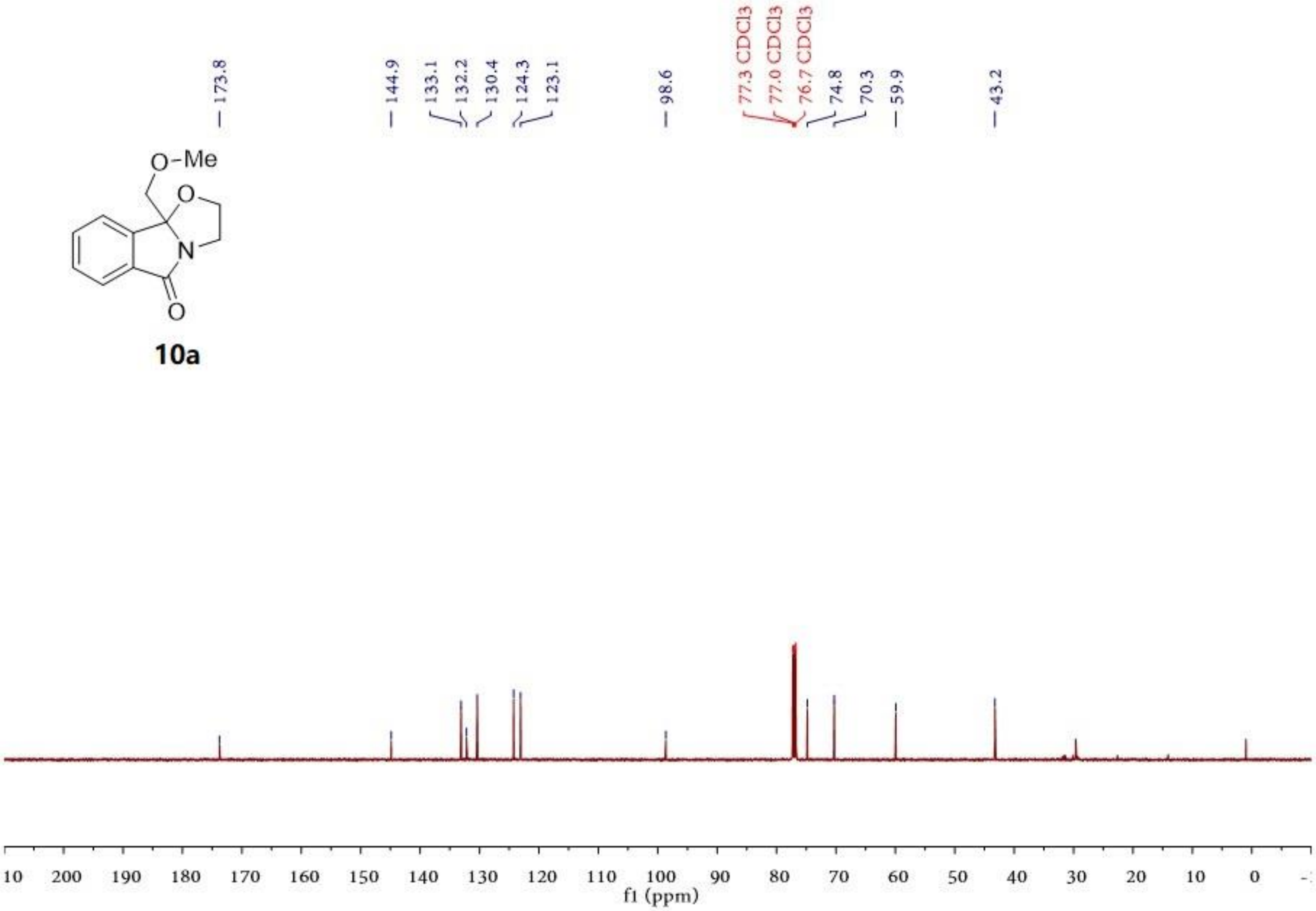
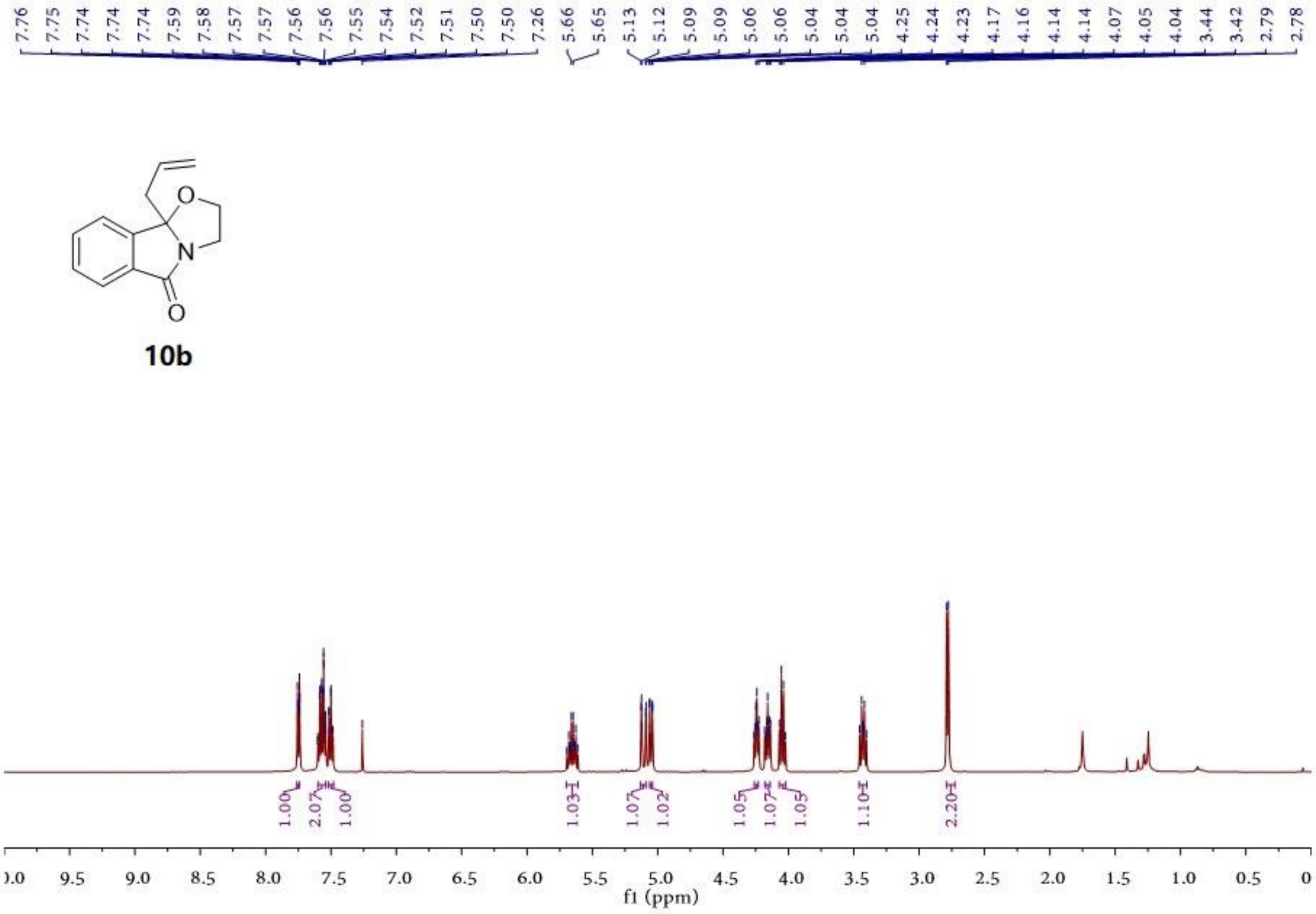
7.59
7.57
7.57
7.54
7.54
7.53
7.52
7.51
7.26
4.33
4.33
4.32
4.31
4.30
4.30
4.17
4.16
4.15
4.14
4.13
4.09
4.07
4.06
3.79
3.77
3.74
3.72
3.52
3.51
3.50
3.49
3.49
3.47
3.43
O–Me
O
N
O
1.00
2.09
1.01
1.07
1.09
1.12
2.19
1.41
3.10
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0
f1 (ppm)

10a


10a

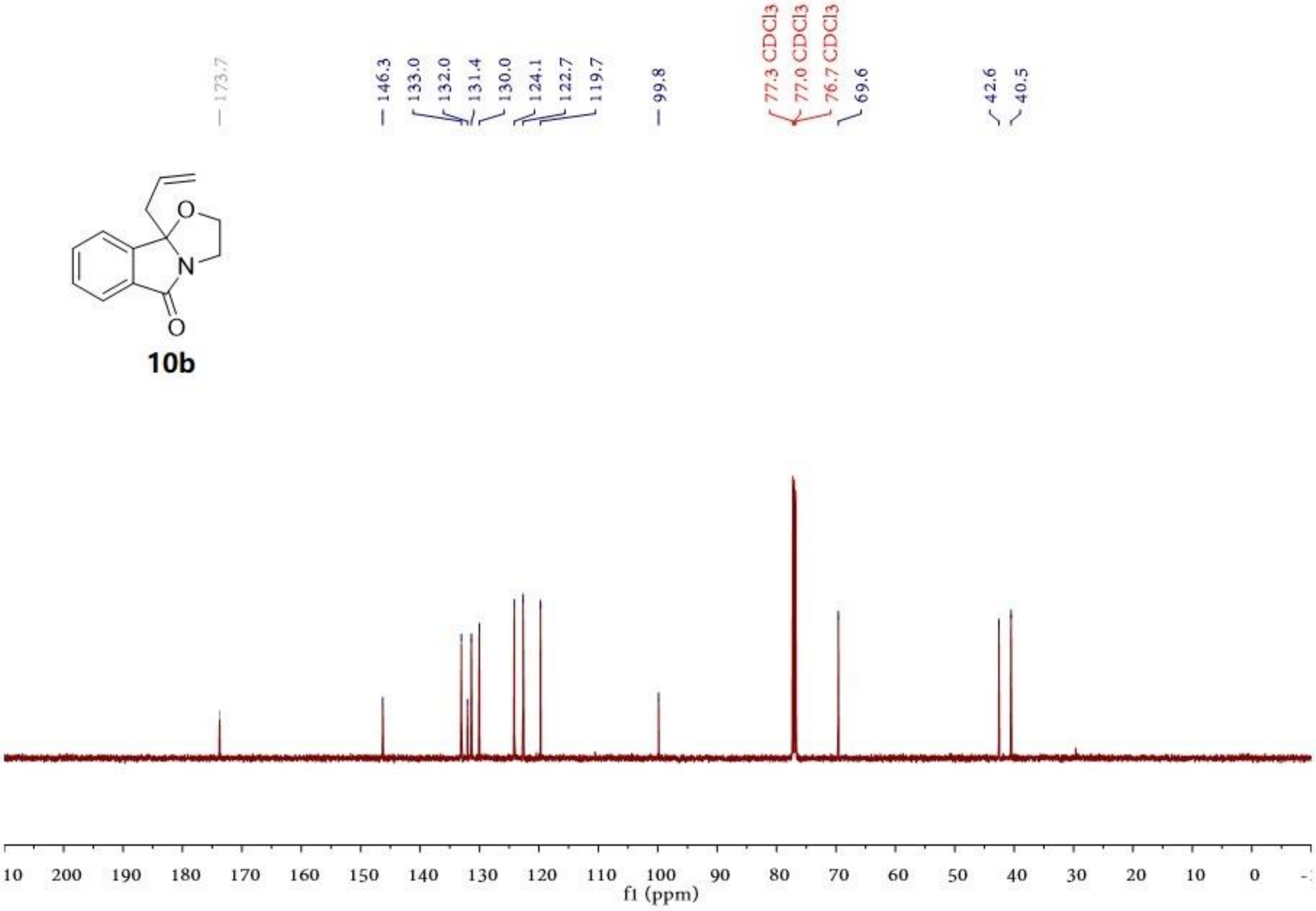
173.8
144.9
133.1
132.2
130.4
124.3
123.1
98.6
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
74.8
70.3
59.9
43.2
O–Me
O
N
O
10
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

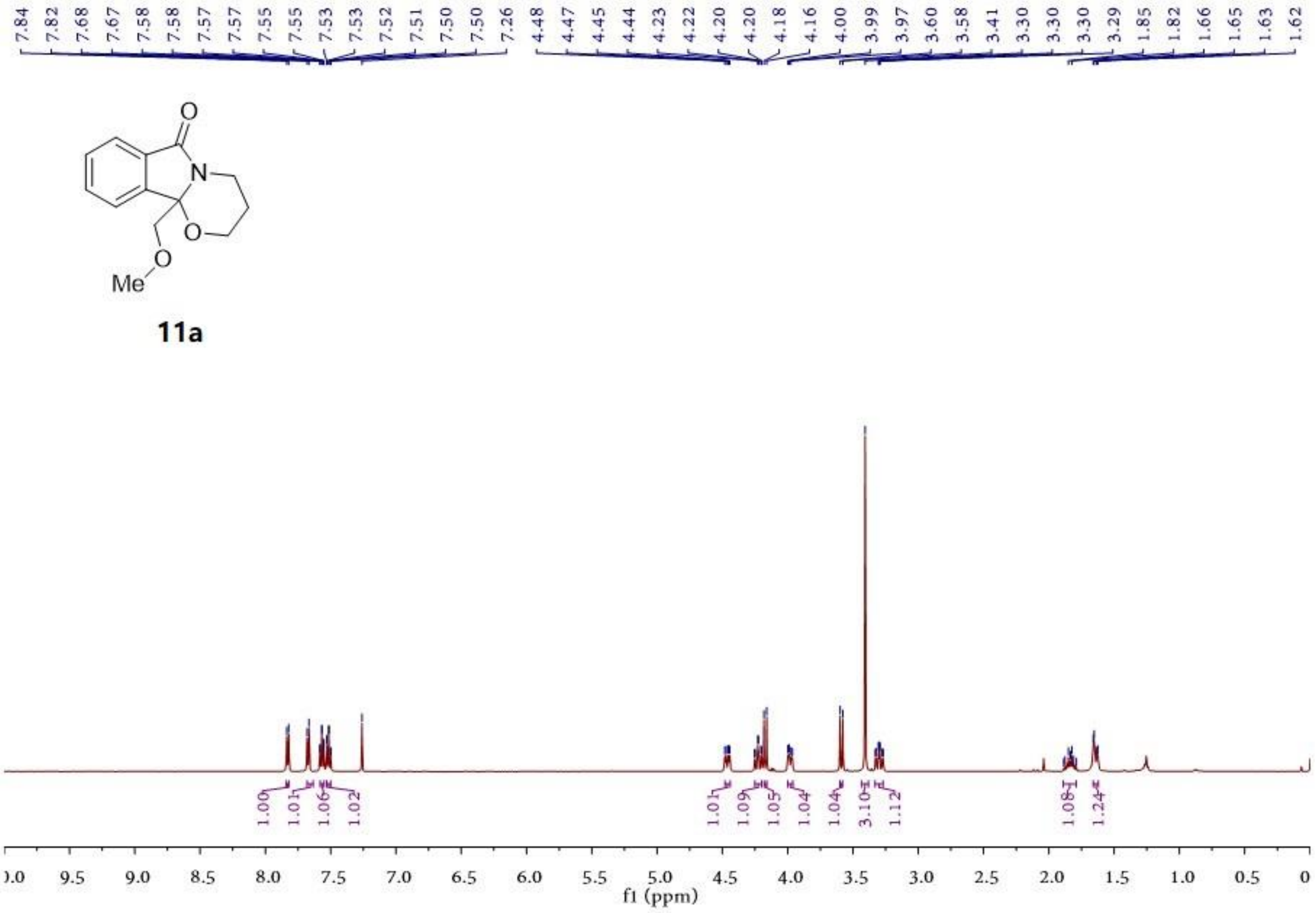

7.76
7.75
7.74
7.74
7.74
7.59
7.58
7.57
7.57
7.56
7.56
7.55
7.54
7.52
7.51
7.50
7.50
7.26
5.66
5.65
5.13
5.12
5.09
5.09
5.06
5.06
5.04
5.04
5.04
4.25
4.24
4.23
4.17
4.16
4.14
4.14
4.07
4.05
4.04
3.44
3.42
2.79
2.78
O
N
O
1.00
2.07
1.00
1.03
1.07
1.02
1.05
1.07
1.05
1.10
2.20
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0
f1 (ppm)

10b

O
N
O

10b


173.7
146.3
133.0
132.0
131.4
130.0
124.1
122.7
119.7
99.8
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
69.6
42.6
40.5
10
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.82
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7.58
7.57
7.57
7.55
7.55
7.53
7.53
7.52
7.51
7.50
7.50
7.26
4.48
4.47
4.45
4.44
4.23
4.22
4.20
4.20
4.18
4.16
4.00
3.99
3.97
3.60
3.58
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3.30
3.30
3.29
1.85
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1.66
1.65
1.63
1.62
O
N
O
O
Me
1.00
1.01
1.06
1.02
1.01
1.09
1.05
1.04
1.04
3.10
1.12
1.08
1.24
).0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
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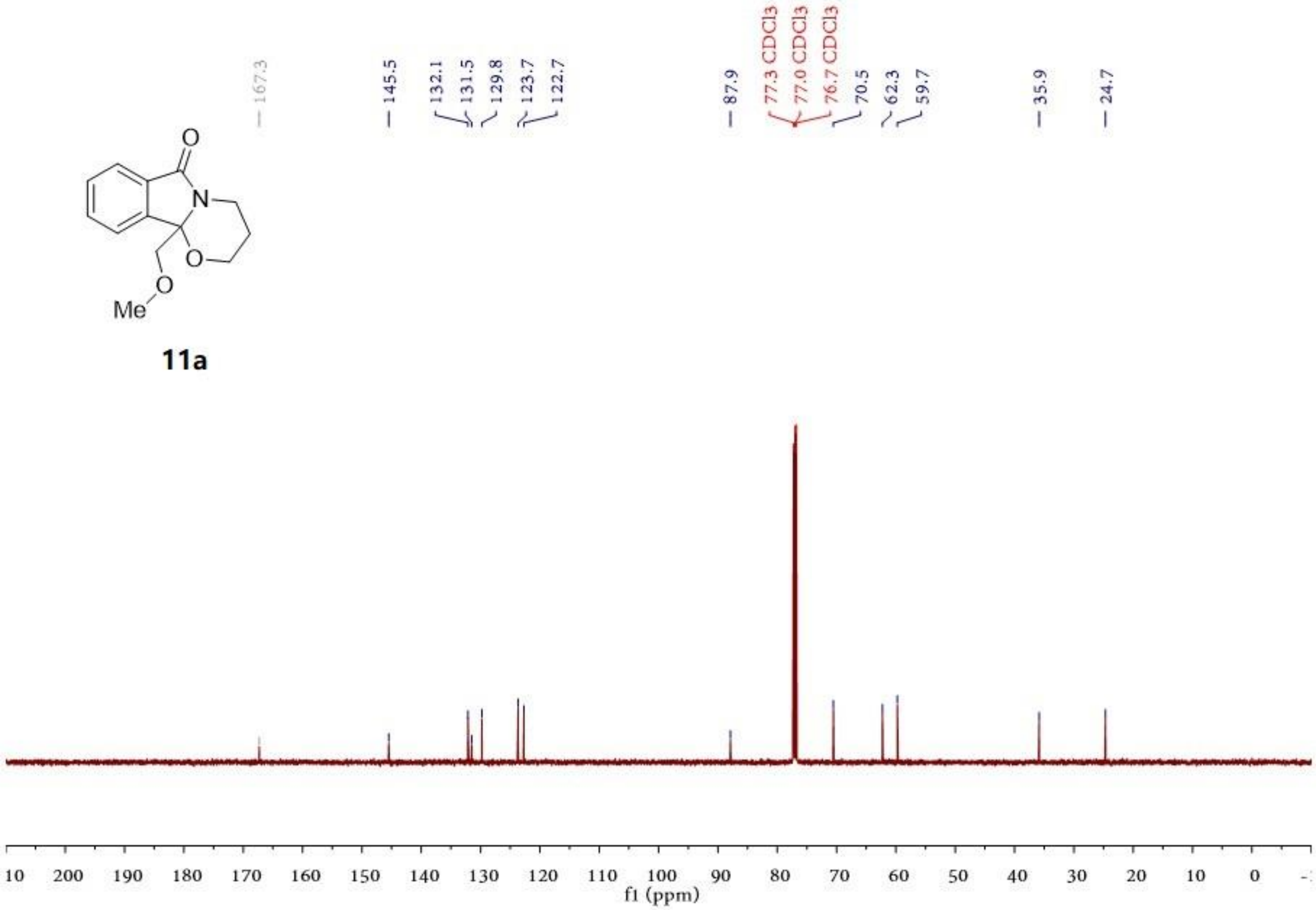
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0
f1 (ppm)

11a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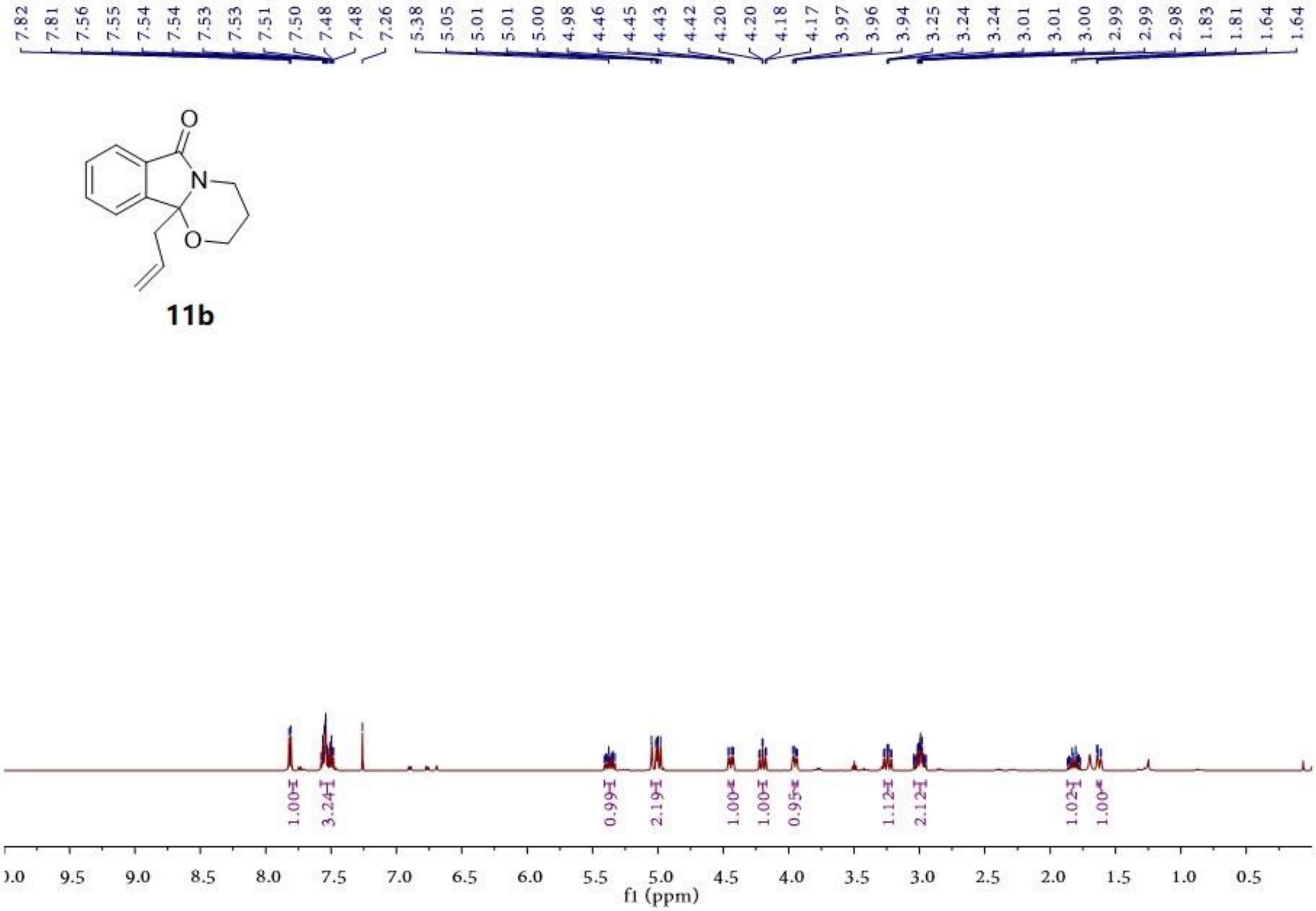
131.5
129.8
123.7
122.7
87.9
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
70.5
62.3
59.7
35.9
24.7
O
N
O
O
Me
10
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

11a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7.26
5.38
5.05
5.01
5.01
5.00
4.98
4.46
4.45
4.43
4.42
4.20
4.20
4.18
4.17
3.97
3.96
3.94
3.25
3.24
3.24
3.01
3.01
3.00
2.99
2.99
2.98
1.83
1.81
1.64
1.64
O
N
O
11b
1.00
3.24
0.99
2.19
1.00
1.00
0.95
1.12
2.12
1.02
1.00
).0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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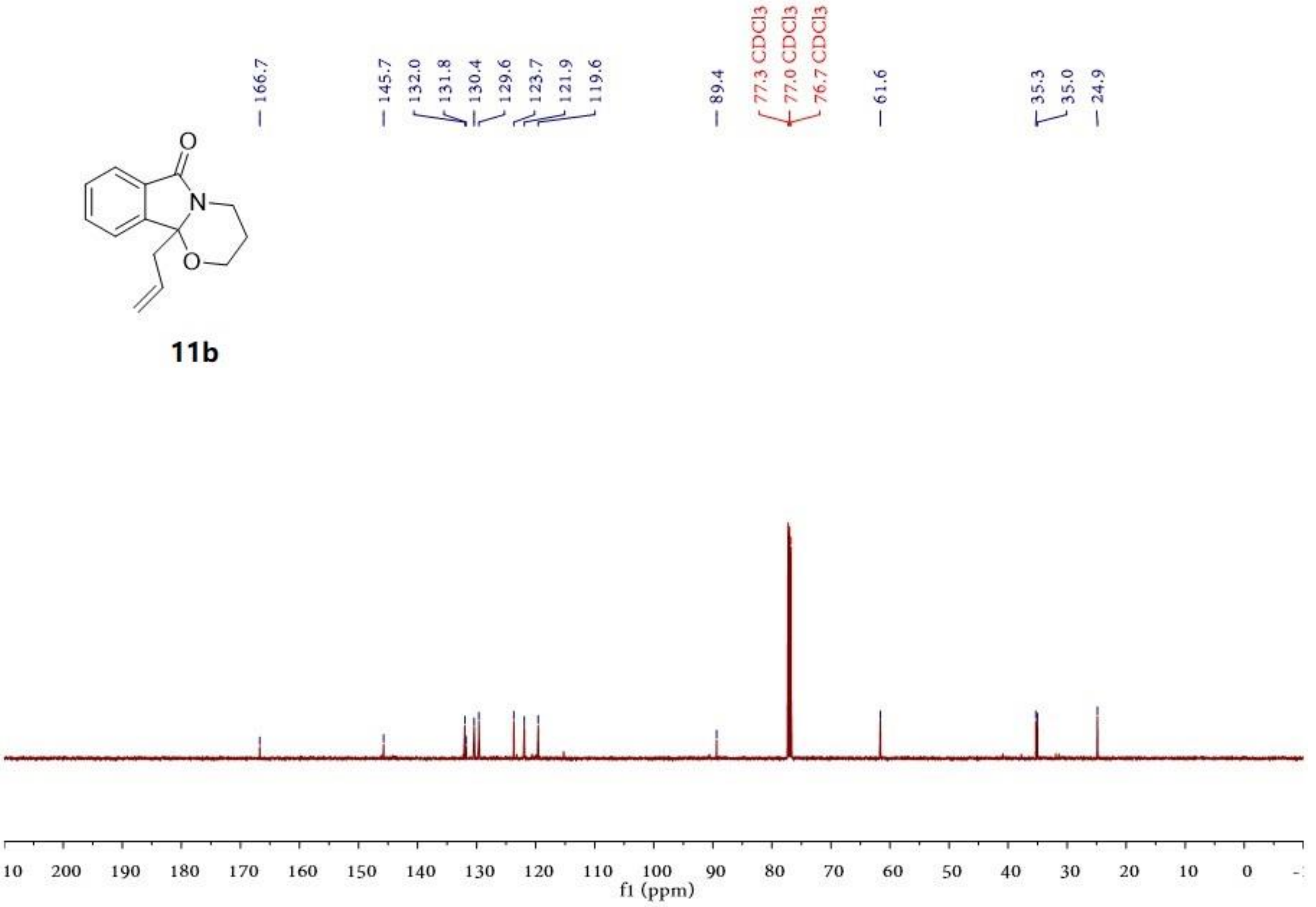
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)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145.7
132.0
131.8
130.4
129.6
123.7
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119.6
89.4
77.3 CDCl3
77.0 CDCl3
76.7 CDCl3
61.6
35.3
35.0
24.9
O
N
O
11b
10
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)